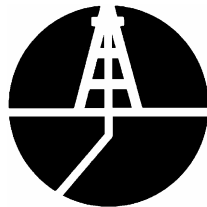


**MMS Project**  
**Long-Term Integrity of Deepwater Cement**  
**Systems Under Stress/Compaction Conditions**

**Report 6**

Issued June 17, 2004

Fred Sabins  
[f.sabins@cementingsolutions.com](mailto:f.sabins@cementingsolutions.com)





## **Table of Contents**

<b>Objectives .....</b>	<b>1</b>
<b>Observations and Recommendations for Future Work .....</b>	<b>1</b>
<b>Testing Program and Procedures .....</b>	<b>1</b>
<i>Cement Design Performance .....</i>	<i>3</i>
<i>Mechanical Properties .....</i>	<i>3</i>
<i>Mechanical Integrity .....</i>	<i>3</i>
<b>Test Results—Mechanical Properties .....</b>	<b>4</b>
<i>Tensile Strength .....</i>	<i>4</i>
<i>Young’s Modulus with Various Confining Forces .....</i>	<i>5</i>
<i>Poisson’s Ratio .....</i>	<i>5</i>
<i>Anelastic Strain .....</i>	<i>6</i>
<b>Test Results—Mechanical Integrity .....</b>	<b>13</b>
<i>Shear Bond .....</i>	<i>13</i>
<i>Annular Seal .....</i>	<i>14</i>
<i>Cement Column Seal .....</i>	<i>24</i>
<b>Appendix A—Test Procedures .....</b>	<b>30</b>
<i>Sample Preparation .....</i>	<i>30</i>
<i>Sample Curing .....</i>	<i>30</i>
<i>Thickening Time Test .....</i>	<i>30</i>
<i>Free-Fluid Test .....</i>	<i>30</i>
<i>Compressive Strength .....</i>	<i>31</i>
<i>Young’s Modulus and Poisson’s Ratio Testing .....</i>	<i>31</i>
<i>Hydrostatic Cycling and Anelastic Strain .....</i>	<i>31</i>
<i>Tensile Strength and Tensile Young’s Modulus .....</i>	<i>32</i>
<i>Annular Seal Testing Procedure .....</i>	<i>33</i>
Soft-Formation Simulation .....	34
Hard-Formation Simulation .....	34
Intermediate Formation Simulation .....	34



<i>Shear Bond Strength Testing .....</i>	<i>35</i>
<i>Cement Column Seal Tests.....</i>	<i>37</i>
<b>Appendix B—Test Data .....</b>	<b>38</b>



## **Objectives**

The overall objective of this project is to determine the properties that affect cement's capability to produce a fluid-tight seal in an annulus. The project primarily focuses on deepwater applications, but general applications will also be examined. The research conducted thus far is focused on the measurement and correlation of cement's mechanical properties to the cement's performance. Also, research was conducted to determine which laboratory methods should be used to establish the cement's key properties.

Results obtained during this reporting period focused on continued measurement of and correlation of cement mechanical properties and mechanical bond integrity of a cemented annulus. Anelastic strain/failure testing results are presented in the Results section below. Mechanical integrity testing included shear bond and annular seal testing on specimens cured under various cyclic curing schedules. The results of these tests are tabulated in the Results section below. Additionally, all test results developed during this project, including graphical data, are presented in Appendix B.

## **Observations and Recommendations for Future Work**

Results of testing during this reporting period indicate:

- Modified Annular Seal and Shear Bond testing performed with the intermediate strength formation was successful and was duplicated for hard and soft formations.
- Analysis of Annular Seal data via Total Energy calculations produced an acceptable method of quantifying the test results.
- Thermal cycling appears to negatively affect foam cement's sealing ability to a greater degree than pressure cycling.
- TXI LightWeight cement performed well in the 8-foot model testing.

Future work includes:

- complete analysis of column sealing tests
- completion of a decision support system (DSS) for optimizing cement composition (the final deliverable of this project)

The DSS will be similar in operation to one commissioned by 3M to select optimum lightweight cement for various conditions. This DSS will accept well conditions as inputs and will contain performance properties for the various cements tested in the project. A semi-quantitative analysis of the inputs vs. cement performance will allow the user to determine the optimum cement composition for maintaining annular seal under the well conditions.

## **Testing Program and Procedures**

The following cement slurries are examined: Type 1, foamed cement, bead cement, Class



H cement, and latex cement. Latex cement designation refers to cements designed with the gas migration control additive D500 which is a microgel type additive. The effects of adding fibers and/or expansion additives to a slurry are also examined. The cements are tested primarily for deepwater applications, but their performance under all application conditions is considered.

Tasks in the project are listed below:

- Task 1 – Problem Analysis
- Task 2 – Property Determination
- Task 3 – Mathematical Analysis
- Task 4 – Testing Baseline
- Task 5 – Refine Procedures
- Task 6 – Composition Matrix
- Task 7 – Conduct Tests
- Task 8 – Analyze Results
- Task 9 – Decision Matrix

Compositions tested in this project are outlined in **Table 1** below. The range of compositions chosen covers the compositions traditionally used in deep water applications as well as newly utilized compositions and compositions designed to produce improved performance.

**Table 1—Cement Compositions for Testing**

Description	Cement	Additives	Water Requirement (gal/sk)	Density (lb/gal)	Yield (ft <sup>3</sup> /sk)
Neat Type I slurry	Type 1	—	5.23	15.6	1.18
Type I slurry with fibers	Type 1	3.5% carbon fibers-milled	5.2	15.6	1.16
Latex slurry	Type 1	1.0 gal/sk LT-D500	4.2	15.63	1.17
Latex slurry with fibers	Type 1	1.0 gal/sk LT-D500 3.5% carbon fibers-milled 0.50% Melkrete	4.09	15.63	1.20
Foam slurry (12-lb/gal)	Type 1	0.03 gal/sk Witcolate 0.01 gal/sk Aromox C-12 1% CaCl	5.2	12.0	1.19
Bead slurry	Type 1	13.19% K-46 beads	6.69	12.0	1.81
Neat Class H slurry	Class H	—	4.3	16.4	1.08
Class H slurry with fibers	Class H	—	4.3	16.4	1.08
Sodium metasilicate slurry	Type 1	—	14.22	12.0	2.40



Four major categories of tests are used to analyze the cements: cement design performance testing, mechanical properties testing, mechanical integrity testing, and numerical simulation. Results of mechanical properties testing and mechanical integrity testing are provided in the “Test Results” section of this report, beginning on Page 4.

### ***Cement Design Performance***

Standard cement design performance testing, including rheology, thickening time, free fluid, set time, compressive strength, etc. are performed according to procedures outlined in API RP 10B.

### ***Mechanical Properties***

Mechanical properties tested include: tensile strength/tensile Young’s modulus (T), compressive Young’s modulus, Poisson’s ratio, and anelastic strain-fatigue testing.

The tensile strengths are determined with the Brazilian Test Method. From this test, the tensile Young’s Modulus is computed, as well as the maximum yield of the sample. By definition, Young’s Modulus is stress applied to the test specimen divided by elastic strain resulting from the stress. Strain is measured in the same direction as applied stress. Tensile Young’s modulus as calculated from these Brazilian Tensile tests is actually a hybrid value since strain is measured in the same direction as applied compressive stress. However, this is orthogonal to resulting tensile stress direction. This accounts for the relatively constantly lower Young’s Modulus determined by this method. The two values are actually related by Poisson’s Ratio.

The compressive Young’s Modulus will be determined through compression tests with confining loads with a baseline of a 14-day cure. Confining loads applied to each composition are varied from 0 psi up to the magnitude of the composition’s compressive failure to determine the affects of confinement on rock properties. Poisson’s ratio will also be determined from these tests. Poisson’s Ratio values will vary with respect to the stress rate, slurry type, air entrainment, and perhaps other variables.

Anelastic strain and fatigue testing is a modification of hydrostatic testing. The modified procedure involves cycling samples repeatedly to 25% or 50% of each composition’s compressive strength under 500-psi confining stress. Measurement of anelastic strain with cycling provides a comparable measure of each composition’s performance.

### ***Mechanical Integrity***

The mechanical integrity issues of the cement slurries include stresses in the cement, and the flow of fluids around the cement and through the matrix of the cement. To predict the flow of fluid around the cement, the cement slurries are tested for bonding capacity, presence of microannuli, and deformation. The flow of fluids through the matrix of the cement is examined through tests for detecting cracks and permeability changes. The stress undertaken by the cement slurries is determined as a function of pressure, temperature, and confining formation strength.



Shear bond and annular seal measurements are taken under cyclical conditions for soft, intermediate strength, and hard formations. Intermediate-strength formation is simulated with Schedule 40 PVC pipe as the outside mold for the cement sheath.

Stresses are imposed on all test specimens by increasing the maximum pressure to which the inner pipe is stressed or by heating the inner pipe. Seal integrity is monitored while stressing the specimens. Additionally, shear bond tests are run only after a composition has been tested for annular seal. The shear bond test samples are subjected to the same pressure and temperature cycling that produced annular seal failure before shear bond is evaluated. This procedure provides a comparison between shear bond and annular seal behavior.

Additional analysis was performed on the complete suite of annular seal data. The analytical method involved measuring sample failure as a function of total work done on the sample by heating or pressure cycling. This analysis revealed a strong relationship between quantity of work applied to a test fixture and failure of the seal.

Cement column seal tests illustrate the sealing effectiveness of several additional cements. These tests are designed to test a cement's capacity to isolate gas pressure across an enclosed column. Eight-foot lengths of 2-in. pipe are filled with cement slurry, pressurized to 1000 psi, and then cured for 8 days. After the test samples have cured, low-pressure gas (100 to 200 psi) is periodically applied to one end of each test pipe and the gas flow rate through the cement column is measured. As time increases with no flow, increased pressure is applied to the pipe to eventually induce failure and flow.

## **Test Results—Mechanical Properties**

This section contains results from testing conducted throughout this project period, as well as additional mechanical property test results selected from previous test periods. Graphical data for all mechanical property tests are presented in Appendix B of this report.

### ***Tensile Strength***

Table 2 shows the effects of carbon fibers on tensile strength. The two-fold to three-fold increase in tensile strength is significant, indicating the potential for fibers to enhance the durability of cement.



**Table 2—Tensile Strength and Tensile Young's Modulus**

Slurry	Tensile Strength (psi)	Young's Modulus
Foam slurry (12-lb/gal)	253	3.23 E4
Neat Type I slurry	394/213 <sup>a</sup>	19.15/8.16 E4 <sup>a</sup>
Type I slurry with fibers	1071	9.6 E4
Latex slurry	539	5.32 E4
Latex slurry with fibers	902	8.5 E4

<sup>a</sup>Data taken from two different specimens.

### ***Young's Modulus with Various Confining Forces***

The effects of confining stress on compressive strength and Young's modulus are presented in Table 3. A significant increase in compressive strength is observed among lower-strength compositions such as foam cement and latex cement, as confining stress is increased.

**Table 3—Young's Modulus at Various Confining Stresses**

Slurry Composition	Confining Pressure (psi)	Young's Modulus (psi)
Type I slurry	0	16.7 E 5
	1500	11.1 E 5
	5000	9.1 E 5
Foam slurry (12 lb/gal)	0	5.8 E 5
	500	6.8 E 5
	1000	6.1 E 5
Bead slurry (12 lb/gal)	0	9.5 E 5
	500	8.1 E 5
	1000	1 E 6
Latex slurry	0	5.6 E 5
	250	8.9 E 5
	500	9.4 E 5

### ***Poisson's Ratio***

Initial results of Poisson's ratio testing on these lightweight cement compositions were unexpectedly low. Continued Poisson's ratio testing confirmed the accuracy of these early results. The low Poisson's ratio values for these compositions are theorized to be related to the porosity of the specimens. Several published technical reports have documented this tendency for Poisson's ratio to be effectively lowered as porosity increases.

Another potential variable in Poisson's ratio testing is load rate. An investigation into the





effect of load rate on Poisson's ratio indicated that load rate does affect Poisson's ratio measurement (Table 4). Testing with Type I Cement at 16.4 lb/gal indicated a decreasing Poisson's ratio with increasing stress rate. A stress rate of 250 psi/min was settled on for remainder of this testing.

**Table 4—Effect of Load Rate on Poisson's Ratio**

<b>Load Rate</b>	<b>Poisson's Ratio</b>
100 psi/min	0.1
250 psi/min	0.08
500 psi/min	-0.01

Table 5 presents data generated with a load rate of 250 psi/min. While these values are lower than what has traditionally been considered acceptable, the data are generally positive. CT scans performed on Poisson's ratio test specimens indicated a link between large voids or pore spaces and variable Poisson's ratio. This procedure will be included in future testing and samples with large voids will be discarded.

**Table 5—Poisson's Ratio  
(50-psi confining pressure, 250 psi/min load rate)**

<b>Slurry</b>	<b>Failure (psi)</b>	<b>Poisson's Ratio</b>
Foam slurry (12-lb/gal)	3100	0.00
Bead slurry	4100	-0.01
Neat Class H slurry	6450	0.0012
SMS slurry	920	0.005
Type I slurry	6500	0.1

### **Anelastic Strain**

Anelastic strain testing is a variation of hydrostatic testing and is designed to allow a more accurate evaluation of permanent strain resulting from stressing different test compositions. This procedure standardizes confining stress at 500 psi and calls for samples to be cycled to 25% and 50% of each composition's compressive strength or failure load under that confining stress. Measurement of anelastic strain with cycling provides a more comparable value of each composition's performance. See Figures 5 and 6.

Results of anelastic strain testing are presented in Table 6. Strain data are reported as final strain minus initial strain measurements, with final being at the end of three cycles. In order to analyze data for different compositions uniformly, a stress point was chosen on the stress-strain plot at a point that the strain appeared to be linear. Strains at this stress magnitude at the beginning and end of cycling were measured and used to calculate plastic deformation. This comparison point is listed also. Data were then normalized with respect to sample length so results appear in units of mm/mm. This step eliminates



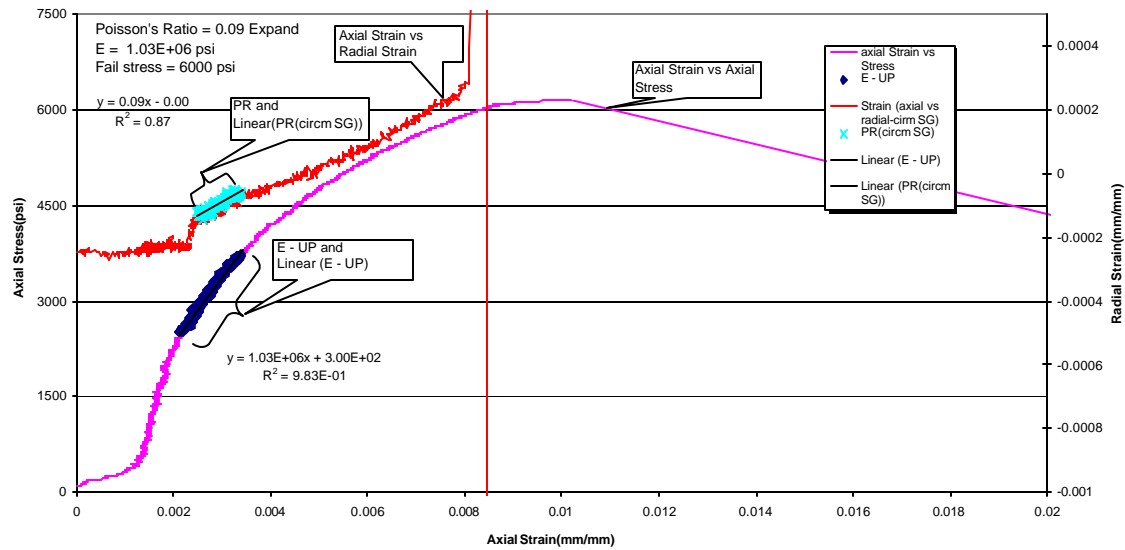
apparent variations in deformation data due to variations in sample size.

**Table 6—Results of Anelastic Strain Testing**

Composition	Failure (psi)	Comparison Stress (psi)	Strain (mm/mm)	
			25%	50%
Type I slurry	6000	600	0.0006	0.0007
Foam slurry	2000	400	0.0009	0.0007
Bead slurry	3300	400	0.0007	0.0005
Latex slurry	6000	600	0.0007	0.0009

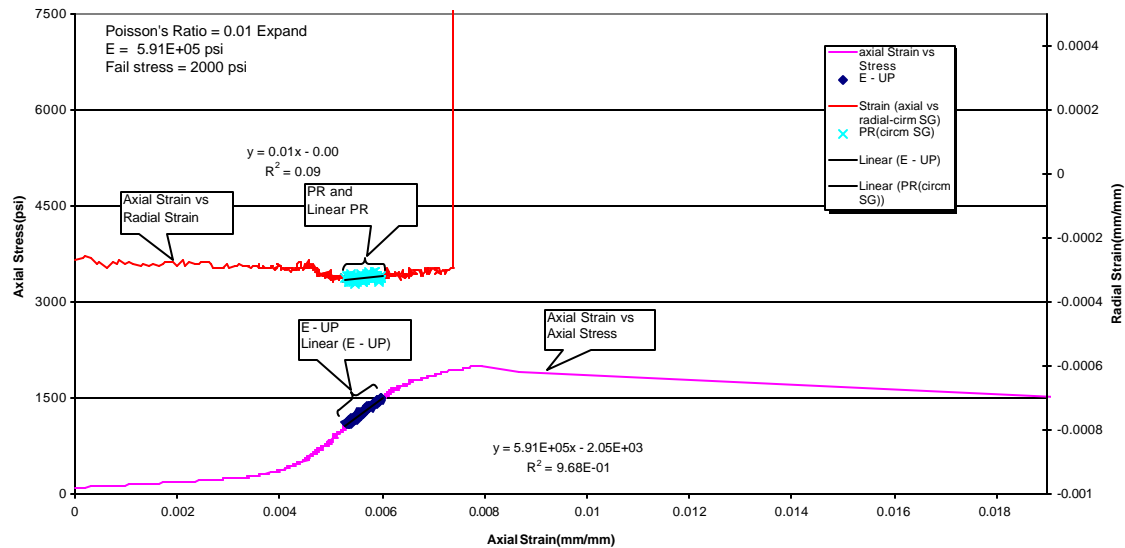
Data generation also includes a round of samples tested to a common stress maximum as seen in Figures 7 through 10 to provide two alternate methods of comparison.

**Figure 1— Anelastic strain failure load for neat Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

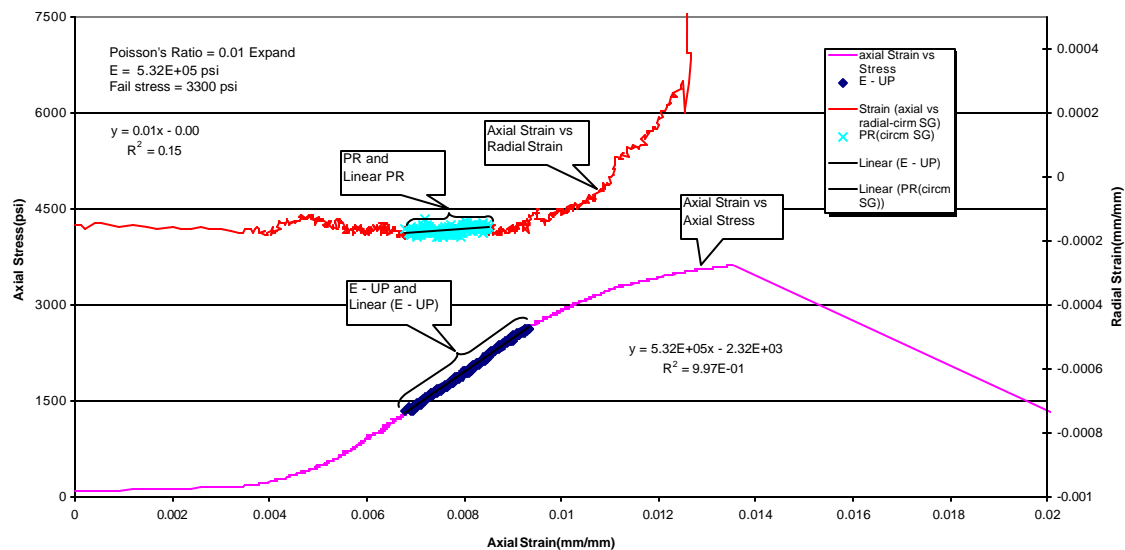




**Figure 2— Anelastic strain failure load for foam slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

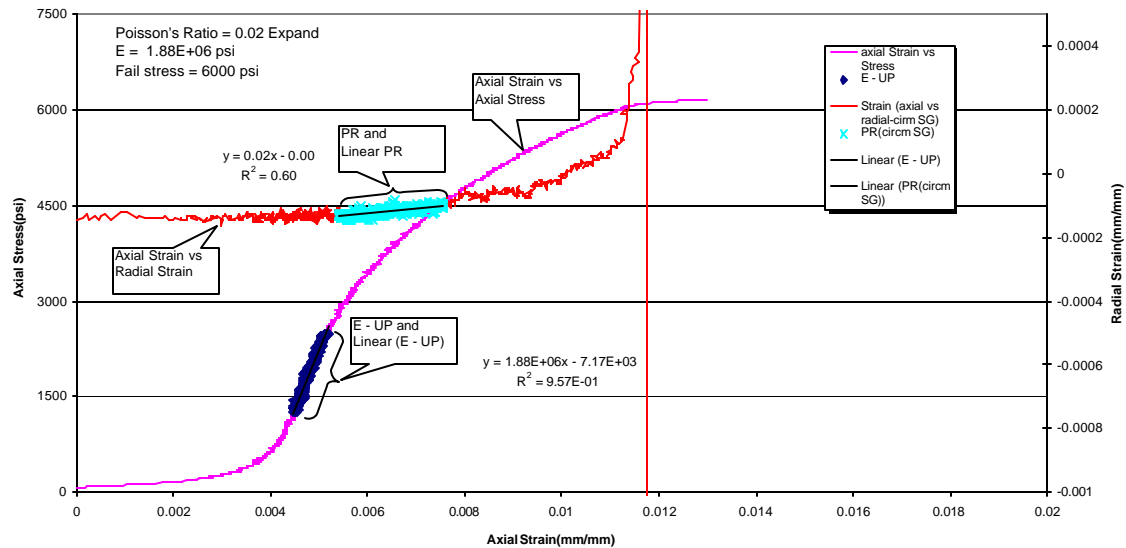


**Figure 3— Anelastic strain failure load for bead slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**





**Figure 4—Anelastic strain failure load for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**



Figures 5 and 6 present strain vs. cycle data for the four compositions at 25% and 50% of each composition's failure stress. Dashed lines represent the slope of each line. Note that all trends are increasing indicating that each specimen would undergo additional anelastic strain with increased cycles. Comparison of the data sets indicates larger strains for low density compositions than for normal density cements.



Figure 5—Anelastic strain comparison of cycles to 25% of failure load

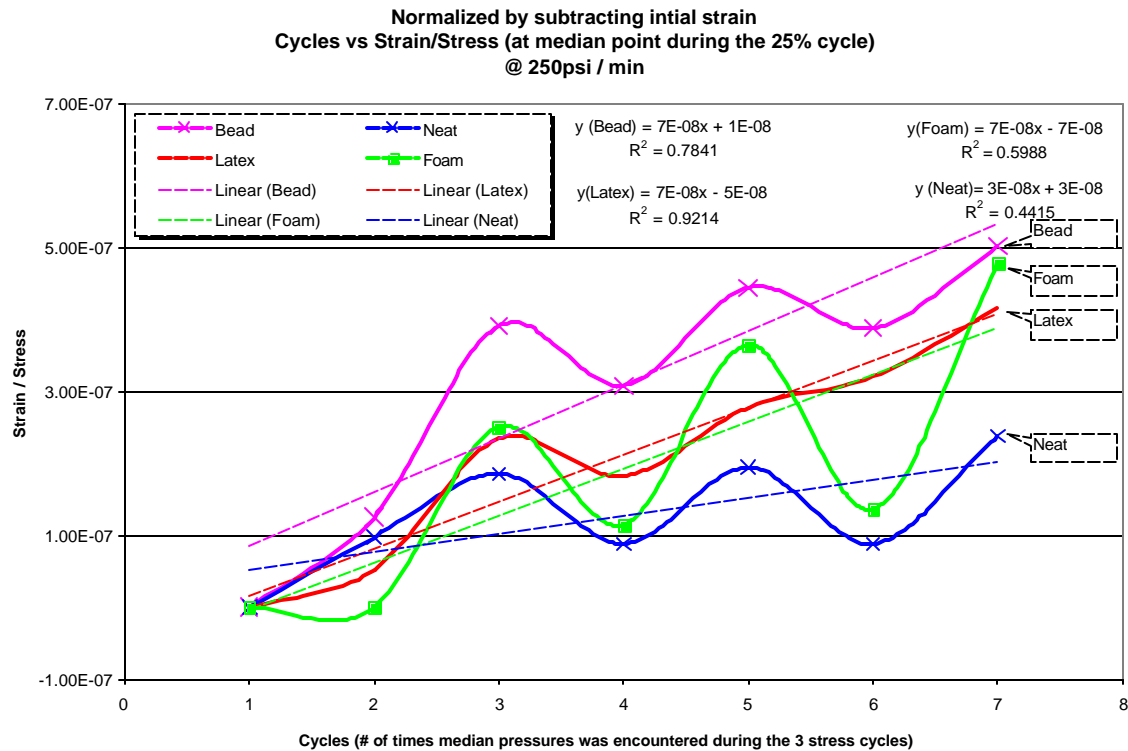
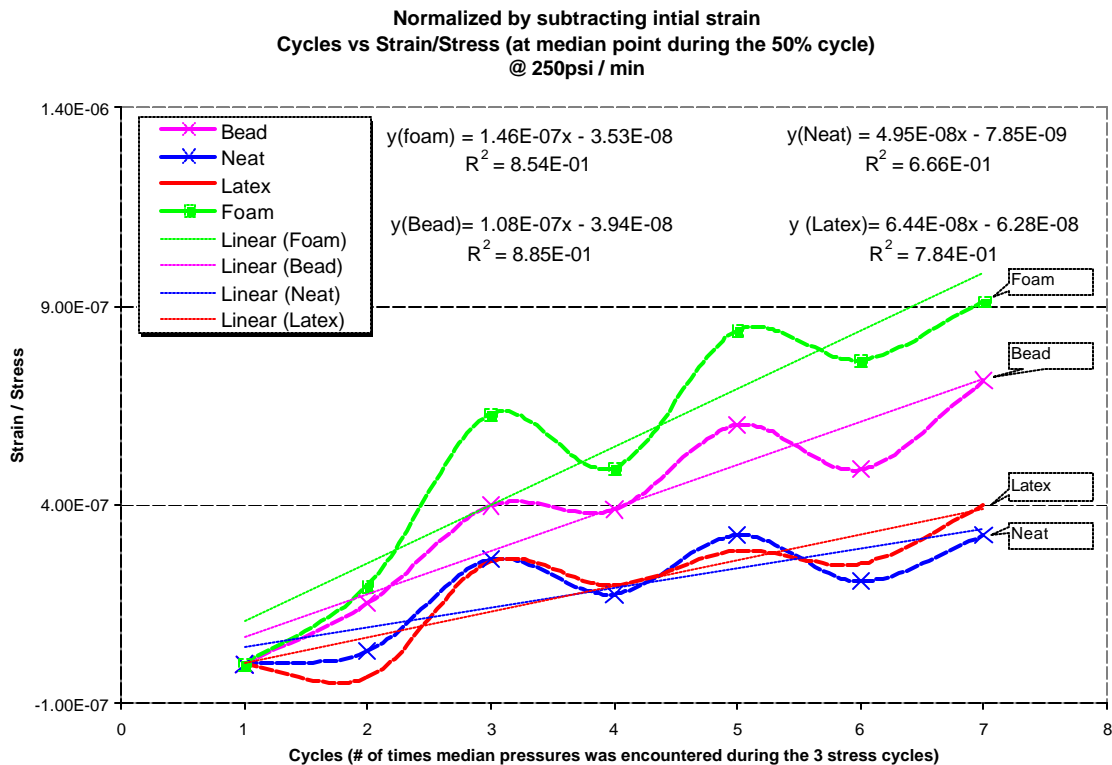


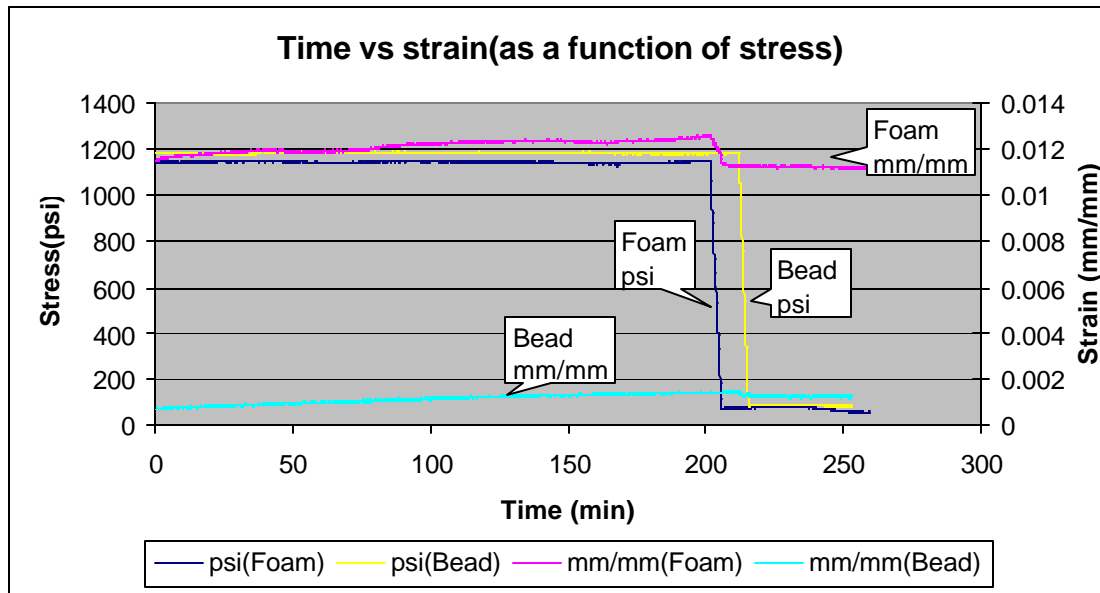
Figure 6—Anelastic strain comparison of cycles to 50% of failure load



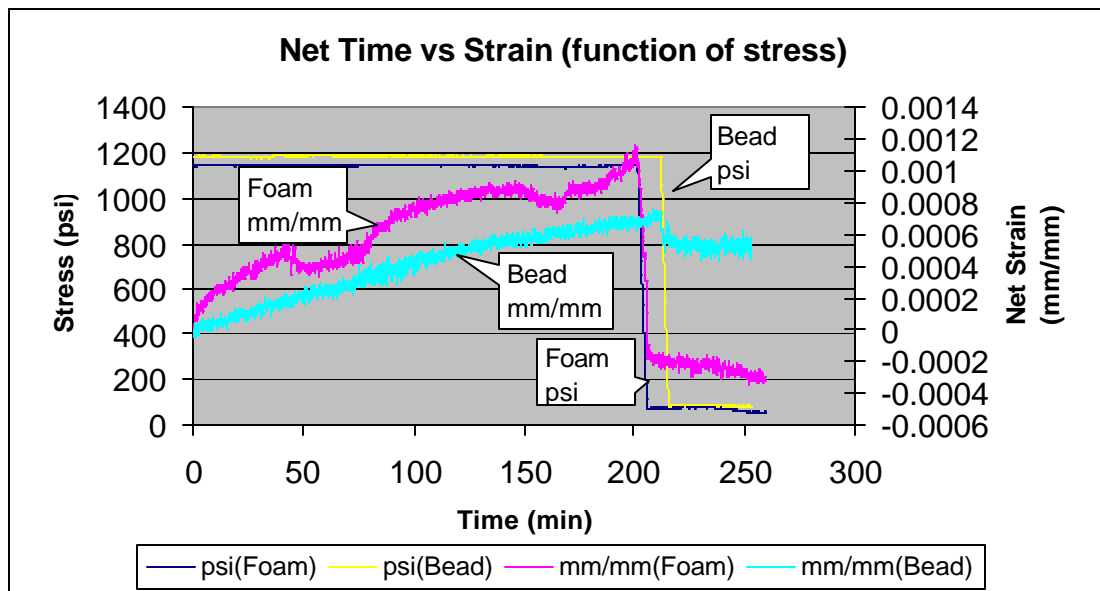


Results of strain vs. time under stress testing are presented in Figures 7 and 8. These results indicate that both foam and bead cement exhibit increasing strain with time under stress. Foam cement's level of strain with increasing stress was slightly more than bead cement.

**Figure 7—Anelastic strain vs. Time comparison of Foam and Bead**



**Figure 8—Anelastic strain comparison of Foam and Bead systems. Strain values from Figure 7 are normalized with respect to each sample's initial strain for comparison.**





Figures 9 and 10 present results of strain measurement vs cyclic stress application. Data from Figure 9 are raw data while those in Figure 10 are normalized with respect to initial strain for each sample. These results indicate significant increase in cycling effect for foam compared to the other three compositions.

**Figure 9—Cyclic Strain comparison of Bead, Foam, Neat and Latex systems**

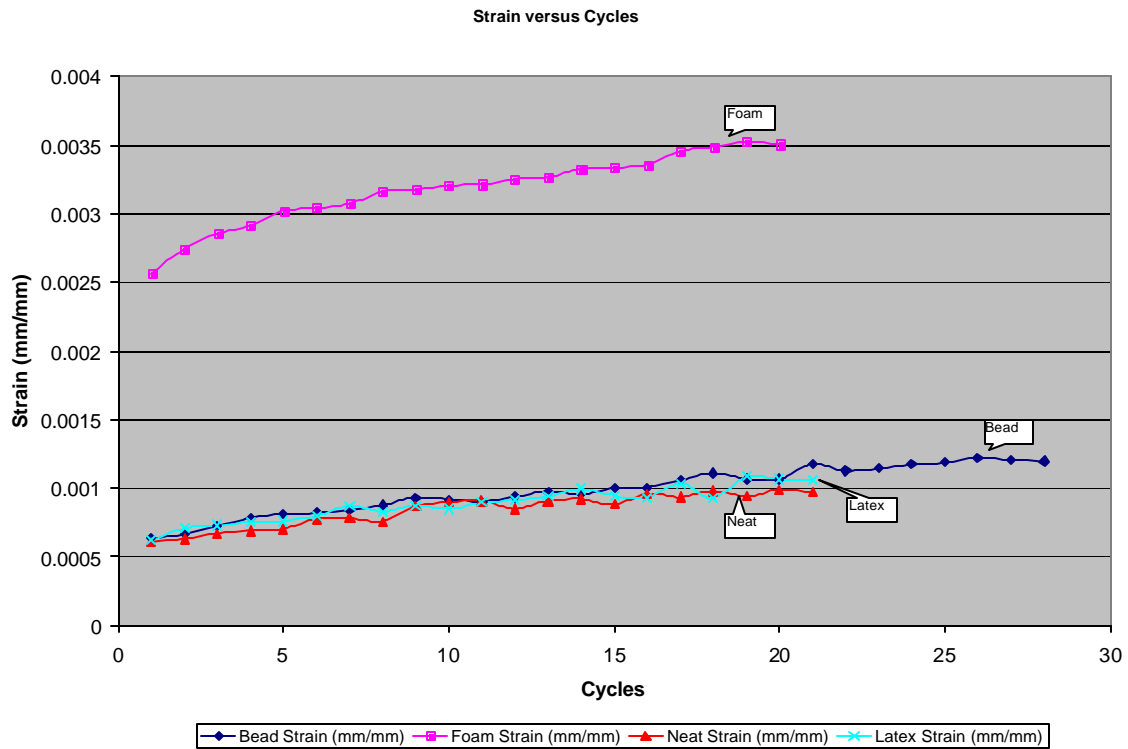
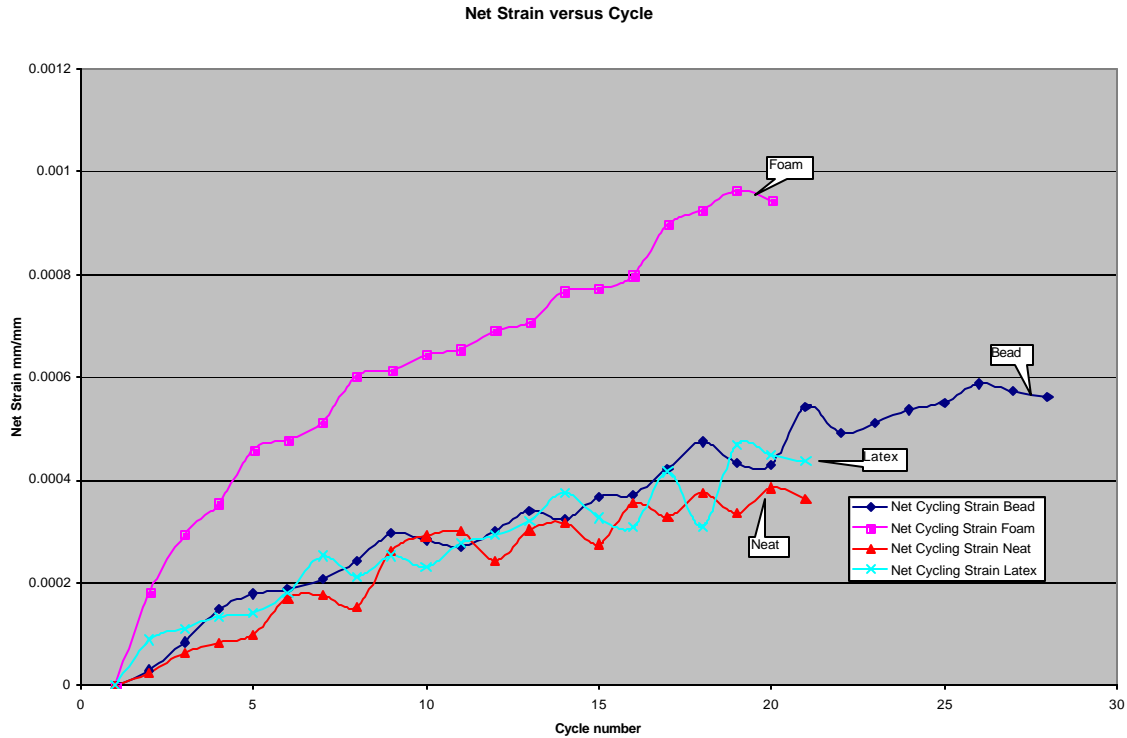




Figure 10—Net Cyclic Strain comparison of Bead, Foam, Neat and Latex systems



## Test Results—Mechanical Integrity

This section contains results from testing conducted throughout this project period, as well as additional mechanical integrity test results selected from previous test periods.

### Shear Bond

Results of shear bond testing (Table 7) indicate that bond is degraded extensively both by pressure and temperature cycling. This degradation seemed to be increased by the presence of simulated soft formation. A modified shear bond method was used with all simulated formations to help ensure that the results are more comparable to annular seal tests (Tables 9, 10, 11 and 13). The test method is explained in Appendix A page 33. Results with hard, intermediate, and soft formations were repeated with the new procedure and results reported in Table 7.





**Table 7—Shear Bond Strengths (psi)**

System	Simulated Formation	Type I Slurry	Foam Slurry	Bead Slurry	Latex Slurry
Baseline	Hard	1228	911	1061	876
	Intermediate	520	298	294	448
	Soft	198	233	143	223
Temperature-Cycled	Hard	293	228	260	244
	Intermediate	209	217	246	194
	Soft	105	44	71	89
Pressure-Cycled	Hard	463	321	386	283
	Intermediate	234	193	192	278
	Soft	141	110	105	84

### **Annular Seal**

Results presented in Table 8 indicate that all cyclic testing specimens failed in the soft formation simulation while all specimens in the hard-formation tests maintained seal. These results indicate the need for a simulated formation with intermediate strength to further differentiate seal effectiveness. Additional stresses for the hard-formation simulation must be imposed through application of heat or pressure.

**Table 8—Annular Seal Tests**

Condition Tested	Formation Simulated	Type I Slurry	Foamed Slurry	Bead Slurry
Initial Flow	Hard	0 Flow	0 Flow	0 Flow
	Soft	0 Flow	0.5 (md)	0 Flow
Temperature-Cycled	Hard	0 Flow	0 Flow	0 Flow
	Soft	0 Flow	123 md	43 md*
Pressure-Cycled	Hard	0 Flow	0 Flow	0 Flow
	Soft	27 md	0.19 md*	3 md

\* Visual inspection revealed samples were cracked.

Modified annular seal testing procedures were employed as outlined in Appendix A page 31 and all three formations including hard, intermediate, and soft were retested using this new procedure. Results for both temperature and pressure cycling are found in Tables 9 through 13. The test methods are explained in Appendix A page 32.

Failure of annular seals was achieved in all formations by increasing cycling until achieving flow. The general trend as can be seen in Tables 9 through 13 was that hard formations needed the greatest amount of cycling to achieve failure. Intermediate formations required less cycling to achieve failure and Soft formations required the least amount of cycling to achieve failure.



Annular seal testing with intermediate-strength formation and increased cyclic loading indicated all materials failed to maintain a seal. Interestingly, foam cement fared best in pressure cycling and worst in temperature cycling.

Table 14 represents a quantifiable measurement of the energy needed whether pressure or temperature induced to produce failure of annular seal. Results of these energy measurements are graphed and compared in Figures 15 and 16.

**Table 9—Annular Seal Pressure-Cycled Slurry Comparison**

Slurry	Form.	Cycle	Pressure (psi)										
			1000-4000	5000	6000	7000	8000	9000	10,000	10,000	10,000	10,000	10,000
Type 1	Hard	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0	0	0
		4	0	0	0	0	0	0	0	0	0.14mD	0.42mD	2.10mD
	Inter.	1	0	0	0	0	0.01 mD	1.1 mD	1.31 mD	2.04 mD	-	-	-
	Soft	1	0	0	0.39 mD	0.39 mD	1.38 mD	+6.69 mD	-	-	-	-	-
Foam	Hard	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0.14mD	0.28mD	0.42mD	1.12mD
	Inter.	1	0	0	0	0	0	0	0	0	0	0	0.79mD
	Soft	1	0	0	0.96 mD	3.2 mD	5.88 mD	+6.4 mD	-	-	-	-	-
Bead	Hard	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0.28mD	1.68mD	2.24mD
	Inter.	1	0	0	0	0	0	0	0.66mD	0.18mD	0.80mD	0.56mD	0.80mD
	Soft	1	0	0	0	0.13 mD	0.39 mD	5.76 mD	+6.4 mD	-	-	-	-
Latex	Hard	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0.03mD	0.14mD	0.28mD	1.4mD	2.1mD
	Inter.	1	0	0	0	0	0.80 mD	2.10 mD	-	-	-	-	-
	Soft	1	0	1.25 mD	+6.4 mD	-	-	-	-	-	-	-	-



**Table 10—Annular Seal Pressure-Cycled Formation Comparison**

Slurry	Form.	Cycle	Pressure (psi)										
			1000-4000	5000	6000	7000	8000	9000	10,000	10,000	10,000	10,000	10,000
Hard	Type 1	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0	0	0
		4	0	0	0	0	0	0	0	0	0.14mD	0.42mD	2.10mD
	Foam	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0.14mD	0.28mD	0.42mD	1.12mD
	Bead	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0.28mD	1.68mD	2.24mD
	Latex	1	0	0	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0.03mD	0.14mD	0.28mD	1.4mD	2.1mD
Interm	Type 1	1	0	0	0	0	0.01 mD	1.1 mD	1.31 mD	2.04 mD	-	-	-
	Foam	1	0	0	0	0	0	0	0	0	0	0	0.79mD
	Bead	1	0	0	0	0	0	0	0.66mD	0.18mD	0.80mD	0.56mD	0.80mD
	Latex	1	0	0	0	0	0.80 mD	2.10 mD	-	-	-	-	-
Soft	Type 1	1	0	0	0.39 mD	0.39 mD	1.38 mD	+6.69 mD	-	-	-	-	-
	Foam	1	0	0	0.96 mD	3.2 mD	5.88 mD	+6.4 mD	-	-	-	-	-
	Bead	1	0	0	0	0.13 mD	0.39 mD	5.76 mD	+6.4 mD	-	-	-	-
	Latex	1	0	1.25 mD	+6.4 mD	-	-	-	-	-	-	-	-



Table 11—Annular Seal Temperature-Cycled Slurry Comparison

Slurry	Form.	Cycles	Temperature Cycles (degrees F)								
			74	94	108	121	135	149	163	176	190
Type 1	Hard	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0
		4	0	0	0	0	0	0	0	0	0
		5	0	0	0	0	0	0.53mD	1.42mD	1.78mD	1.78mD
	Interm.	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	2.89mD	3.34mD	5.78mD	-	-
	Soft	1	0	0	0	0	0	0	0	0	0
		2	0	0	1.23mD	1.63mD	1.63mD	7.98mD	+8.16mD	-	-
Foam	Hard	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0.71mD	1.07mD	2.67mD	3.56mD	4.45mD
	Interm.	1	0	0	0	0.07mD	0.22mD	1.22mD	-	-	-
	Soft	1	0	0.49mD	0.65mD	0.98mD	1.21mD	1.31mD	1.31mD	1.31mD	+8.16mD
Bead	Hard	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	1.78mD	3.56mD	5.34mD	8.90mD	-	-	-
	Interm.	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	3.11mD	3.71mD	-
	Soft	1	0	0	0	0	0	0	0	0	0
		2	0	0	0.41mD	2.45mD	+8.16mD	-	-	-	-
Latex	Hard	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0
		4	0	0	0	0	0	0.89mD	2.31mD	2.67mD	3.56mD
	Interm.	1	0	0	0	0	0.01mD	0.93mD	1.33mD	3.34mD	-
	Soft	1	0	0	0	0.82mD	1.01mD	1.14mD	1.24mD	1.96mD	+8.16mD

Failure of the cement sheath in a wellbore environment is due to imposed stresses that are greater than the cement can withstand. Measurement of stresses becomes difficult, even in laboratory models because of the non-homogeneous composite nature of the cement itself. Specifically, the different types of cements contribute to the difficulty, because of the very different ways in which they respond to applied pressure and temperature loads. While pressure loads can be related to gross stress relatively simply, the effect of temperature is problematic due to the complex wellbore geometry and the many and variable system constraints. To address this difficulty and quantify performance of the various test compositions in the annular seal model, failure was related to the total energy input to the wellbore / cement / formation system. Energy input is in one of two forms, pressure or temperature. Ultimately, the stresses imposed are caused by the input of energy to the system. This simplification bypasses the problem of the non-uniform distribution of these stresses in the non-homogeneous material.

The correlation of energy input to ultimate cement failure was done in order to better understand the mechanisms associated with wellbore cement integrity. The results of this correlation are presented in Tables 12 through 14 and Figures 11 through 16. Further work is required to fully understand the mechanisms by which hydraulic or thermal



energy ultimately leads to cement failure. In the current small sample, the following observations are offered:

- With only two exceptions, the amount of energy (pressure or temperature) required to induce cement sheath failure increases with the competence of the formation. The stronger the formation, the more support it lends to the cement sheath so that it can withstand the imposed loads.
  - The two exceptions involve the temperature energy applied to Bead systems. In these cases, the energy to initiate failure is slightly higher in the intermediate formation than the hard, although statistically they may be equivalent. The explanation is that in the case of temperature, the superior insulating properties of the beads reduce the importance of formation competence, within limits. This represents an important finding supporting the use of beads in cases that may traditionally have indicated foam. The stronger encapsulation of the air pocket in bead vs foam means that the bead cements will withstand heat better than foam systems.
- Bead cements performed very well in all the testing, as evidenced in the cases of weaker formations. In the case of pressure energy, foam also performed better than Type 1 and Latex slurries with weaker formation support. This may be due to better anelastic behavior, in which the cement is more ductile than the higher-strength systems.
- In all cases, the amount of temperature energy required to initiate failure is much lower than the pressure energy to failure. The reason for this is believed to be the destructive effects of matrix water expansion with temperature.
- At this point, with limited data, the results cannot be scaled up from lab to field geometries with confidence. More work is required to understand the energy absorption of the various wellbore components, so that the energy applied to the slurry itself is isolated and understood. As a qualitative example, heavier wall internal pipe will absorb more energy, thereby reducing the energy input to the slurry. More testing will allow in-depth understanding of energy distribution in the wellbore.



**Table 12—Dissipated Energy to Failure**

**Results Summary**

Dissipated Energy to failure

*Pressure Results*

Joules / cu in  
cement

	Formation		
	Hard	Intermed	Soft
Cement			
Bead	741	131	61
Foam	436	247	44
Latex	683	81	29
Type 1	1,017	81	44

Joules / lbm  
cement

	Formation		
	Hard	Intermed	Soft
Cement			
Bead	14,269	2,518	1,175
Foam	8,393	4,756	839
Latex	10,096	1,203	430
Type 1	15,065	1,205	646

*Temperature*

*Results*

Joules / cu in  
cement

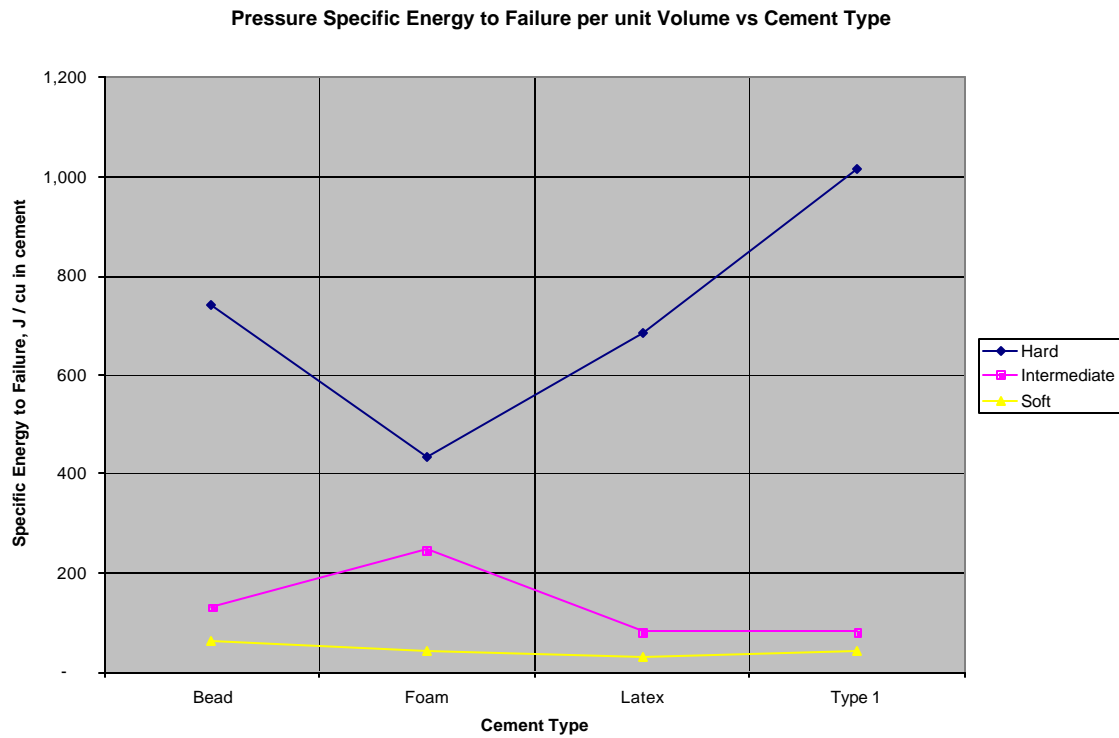
	Formation		
	Hard	Intermed	Soft
Cement			
Bead	283	316	170
Foam	186	65	44
Latex	421	72	65
Type 1	535	186	170

Joules / lbm  
cement

	Formation		
	Hard	Intermed	Soft
Cement			
Bead	5,453	6,085	3,267
Foam	3,578	1,242	851
Latex	6,227	1,069	954
Type 1	7,920	2,752	2,513



**Figure 11—Pressure Specific Energy to Failure per unit Volume vs Cement Type**



**Figure 12—Pressure Specific Energy to Failure per unit Mass vs Cement Type**

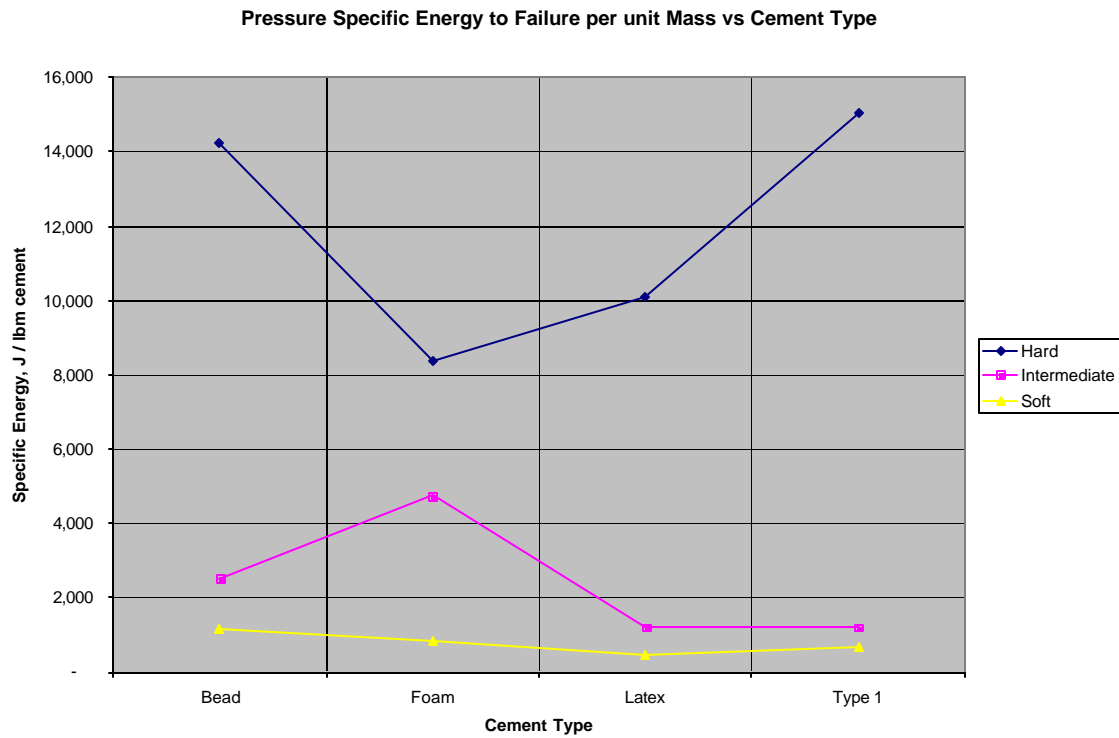




Figure 13—Temp. Specific Energy to Failure per unit Volume vs Cement Type

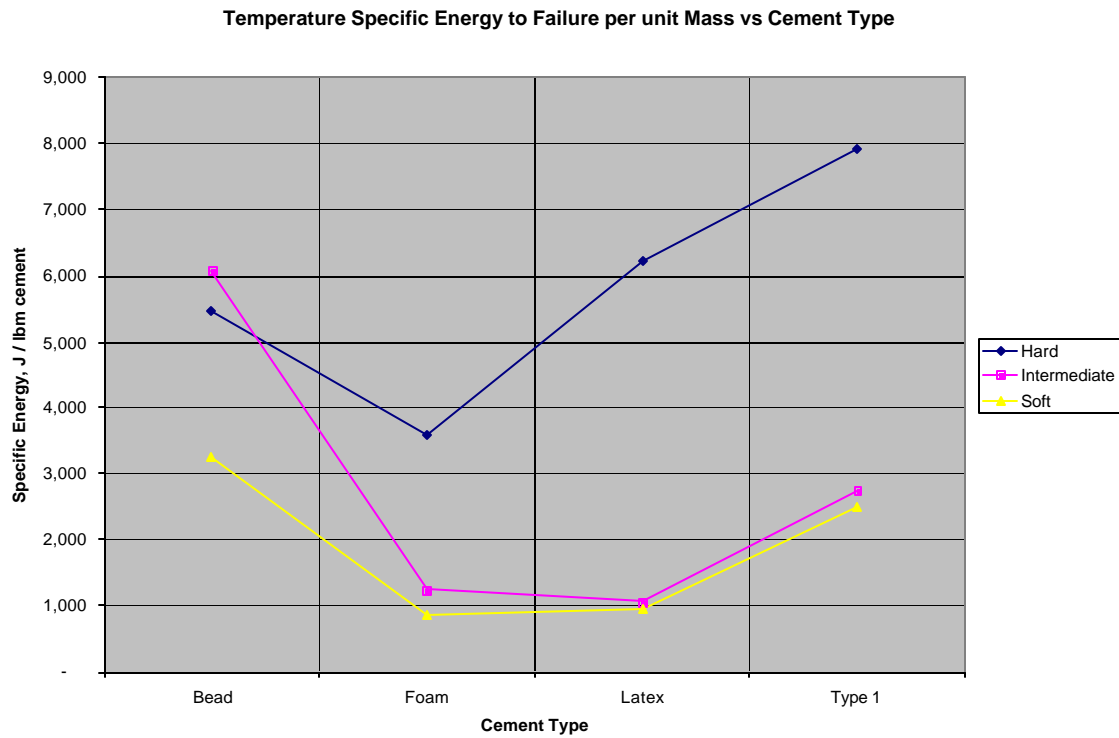


Figure 14—Temp. Specific Energy to Failure per unit Mass vs Cement Type

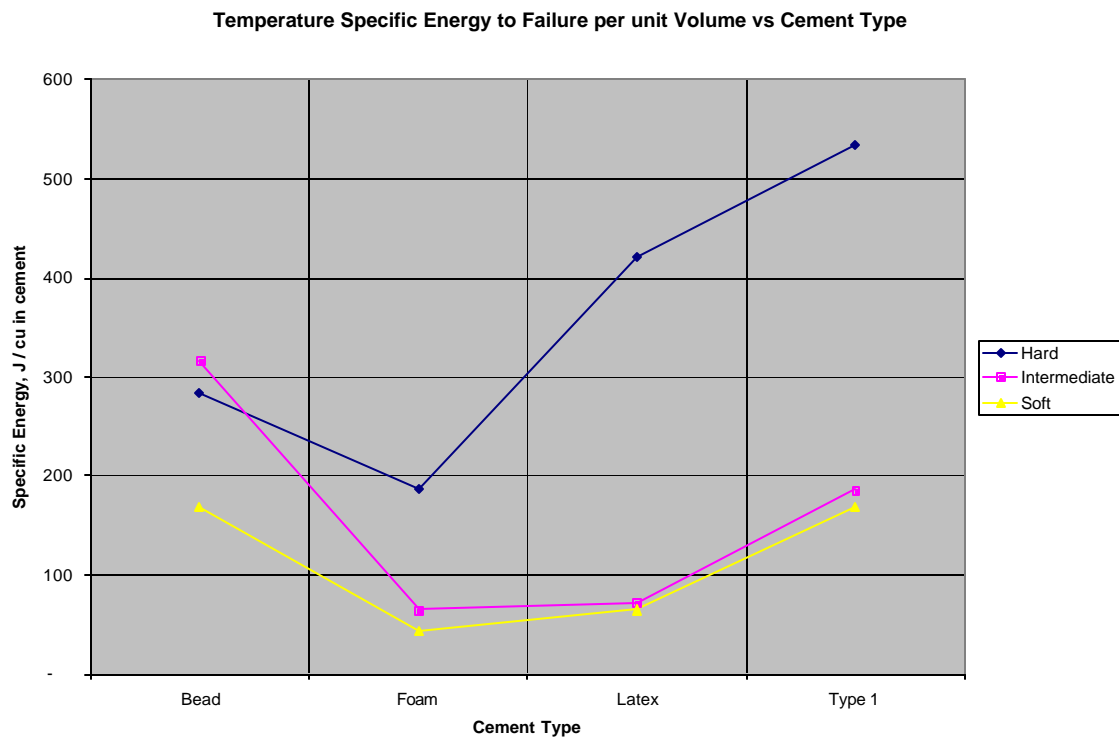






Table 13—Annular Seal Temperature-Cycled Formation Comparison

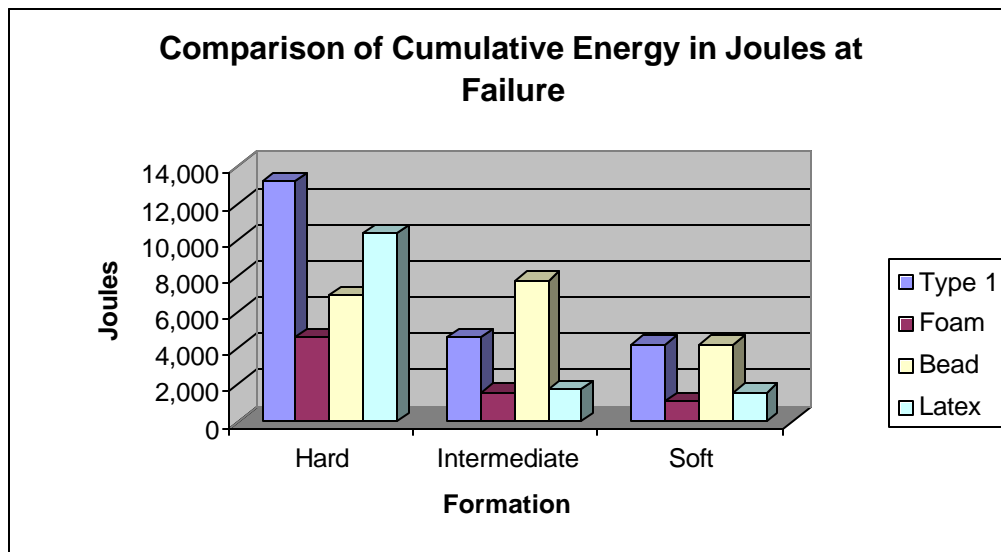
Slurry	Form.	Cycles	Temperature Cycles (degrees F)								
			74	94	108	121	135	149	163	176	190
Hard	Type 1	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0
		4	0	0	0	0	0	0	0	0	0
		5	0	0	0	0	0	0.53mD	1.42mD	1.78mD	1.78mD
	Foam	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0.71mD	1.07mD	2.67mD	3.56mD	4.45mD
	Bead	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	1.78mD	3.56mD	5.34mD	8.90mD	-	-	-
	Latex	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
		3	0	0	0	0	0	0	0	0	0
		4	0	0	0	0	0	0.89mD	2.31mD	2.67mD	3.56mD
Interm	Type 1	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	2.89mD	3.34mD	5.78mD	-	-
	Foam	1	0	0	0	0.07mD	0.22mD	1.22mD	-	-	-
		2	0	0	0	0	0	0	0	0	0
	Bead	1	0	0	0	0	0	0	0	0	0
		2	0	0	0	0	0	0	0	0	0
	Latex	1	0	0	0	0	0.01mD	0.93mD	1.33mD	3.34mD	-
Soft	Type 1	1	0	0	0	0	0	0	0	0	0
		2	0	0	1.23mD	1.63mD	1.63mD	7.98mD	+8.16mD	-	-
	Foam	1	0	0.49mD	0.65mD	0.98mD	1.21mD	1.31mD	1.31mD	1.31mD	+8.16mD
	Bead	1	0	0	0	0	0	0	0	0	0
		2	0	0	0.41mD	2.45mD	+8.16mD	-	-	-	-
	Latex	1	0	0	0	0.82mD	1.01mD	1.14mD	1.24mD	1.96mD	+8.16mD



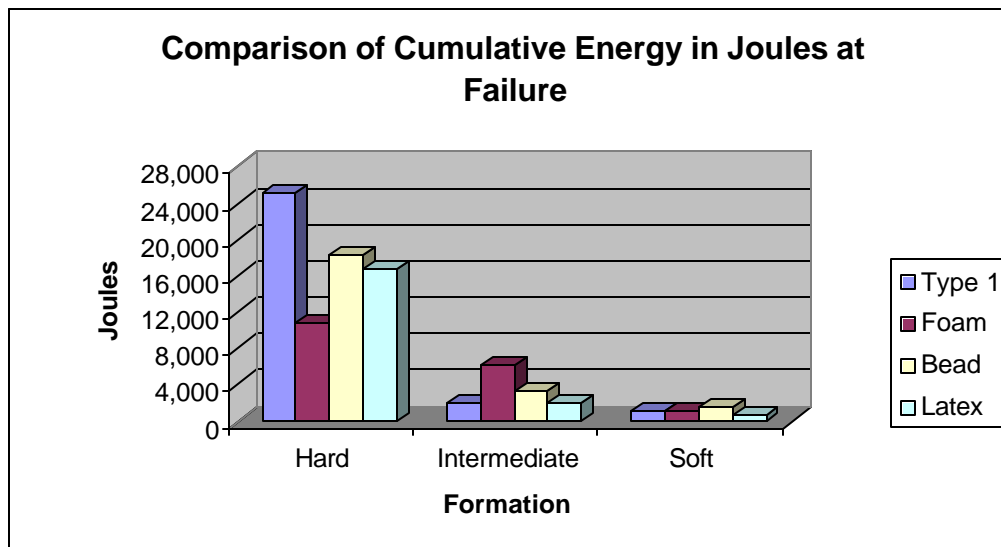
**Table 14—Annular Seal Cumulative Energy at Failure (Joules)**

	Type 1		Foam		Bead		Latex	
Formation	Temp.-Cycled	Press.-Cycled	Temp.-Cycled	Press.-Cycled	Temp.-Cycled	Press.-Cycled	Temp.-Cycled	Press.-Cycled
Hard	13,226	25,157	4,596	10,782	7,004	18,329	10,418	16,891
Intermediate	4,596	2,013	1,596	6,110	7,817	3,235	1,788	2,013
Soft	4,197	1,078	1,094	1,078	4,197	1,509	1,596	719

**Figure 15—Annular Seal Failure for Temperature-Cycled**



**Figure 16—Annular Seal Failure for Pressure-Cycled**





### Cement Column Seal

Four duplicate sets of models were filled with cement compositions listed in Table 15.

**Table 15—Compositions Tested for 8-ft Permeability Models**

Composition	Density (lb/gal)	Yield (ft <sup>3</sup> /sk)	Water (gal/sk)	Columns
Type I slurry	15.6	1.18	5.23	1 and 2
SMS slurry	12	2.38	14.05	3 and 4
Bead slurry	12	1.81	6.69	5 and 6
Latex slurry	15.63	1.17	4.20	7 and 8

These cements were allowed to cure for 7 days, and were then tested with differential pressure as described in the procedure section. Results, summarized in Table 16, are for days tested after the initial curing period. Actual results are shown in Appendix B, Table B1, page 54.

**Table 16—Failure of 8-ft Permeability Models**

Column	Days Tested until Failure	Pressure Differential (psi)	Permeability (mD)
1	107	500	0.09
2	51	200	0.1
3	1	100	33
4	1	100	26
5	78	400	0.03
6	84	400	0.02
7	84	400	0.02
8	99	500	3.1

These results indicate that the sodium metasilicate (SMS) cement failed very quickly on the first day of testing. Other compositions including the neat Type 1 cement required up to 500 psi over the 8-ft column to induce failure.

A second set of 8ft. Permeability models were filled with cement compositions listed in Table 17. These compositions were selected to represent a range of materials that could be formulated from conventional light-weight additives. Density ranges from 12 to 13 lb/gal were tested to determine at what density each additive might produce a successfully-sealing cement.

**Table 17—Compositions Tested for second set of 8-ft Permeability Models**

<b>Composition</b>	<b>Density (lb/gal)</b>	<b>Yield (ft<sup>3</sup>/sk)</b>	<b>Water (gal/sk)</b>	<b>Columns</b>
Type I slurry with 20% Gel	12.0	2.77	16.24	1
Type I slurry with 18% Gel	12.5	2.4	13.56	2
Type I slurry with 16% Gel	13.0	2.11	11.47	3
Type I slurry with 3% SMS	12.5	2.11	12.05	4
Type I slurry with 2.5% SMS	13.0	1.88	10.32	5
65:35 TypeI:Poz slurry with 16% Gel	12.0	1.79	10.11	6
65:35 TypeI:Poz slurry with 12% Gel	12.5	1.38	7.12	7
65:35 TypeI:Poz slurry with 10% Gel	13.0	2.4	13.71	8
TXI LW slurry with 2% SMS	12.0	2.04	11.19	9
Neat TXI LW slurry	13.0	1.79	9.4	10

These cements were allowed to cure for 3 days, and were then tested with differential pressure as described in the procedure section. Results, summarized in Table 18 are for days tested after the initial curing period. Actual results are shown in Appendix B, Table B2, page 55.

Results from Table 18 indicate that a seal was maintained for 13-lb/gal gel and sodium silicate cements. No formula with pozzolan maintained a seal while both TXI LightWeight cements maintained seals.



**Table 18---Failure of second set of 8-ft Permeability Models**

Column	Days Tested at 100 psi	Permeability (mD)
1	1	2.41
2	3	1.23
3	90	0
4	3	2.29
5	90	0
6	1	6.73
7	1	0.89
8	25	0.38
9	90	0
10	90	0

A third set of 8ft. Permeability models were filled with cement compositions listed in Table 19. These compositions were selected to represent an additional range of materials that could be formulated from conventional light-weight additives. Density ranges from 11 to 13.5 lb/gal were tested to determine at what density each additive might produce a successfully-sealing cement.

**Table 19—Compositions Tested for third set of 8-ft Permeability Models**

<b>Composition</b>	<b>Density (lb/gal)</b>	<b>Yield (ft<sup>3</sup>/sk)</b>	<b>Water (gal/sk)</b>	<b>Columns</b>
Type I slurry with 2% SMS	13.4	1.72	9.17	1
Type I slurry with 2% SMS	13.0	1.87	10.28	2
TXI LW slurry with 3% SMS	11.0	2.49	15.30	3
TXI LW slurry with 3% SMS	11.5	2.10	12.35	4
65:35 TypeI:Poz slurry with 6% Gel	13.5	1.56	7.84	5
50:50 TypeI:Poz slurry with 6% Gel	13.4	1.51	7.46	6
50:50 TypeI:Poz slurry with 8% Gel	12.8	1.75	9.14	7
50:50 TypeI:Poz slurry with 10% Gel	12.4	1.95	10.61	8
TXI H slurry with 12% Gel	12.0	2.60	15.30	9
TXI H slurry with 8% Gel	12.5	2.21	12.58	10

These cements were allowed to cure for 3 days, and were then tested with differential pressure as described in the procedure section. Results, summarized in Table 20 are for days tested after the initial curing period. Actual results are shown in Appendix B, Table B3, page 56.



Results from Table 20 indicate that a seal was maintained for the 65:35 Typel:Poz slurry with 6% Gel mixed at 13.5-lb/gal. All other formulations did not maintain seals.

**Table 20---Failure of third set of 8-ft Permeability Models**

<b>Column</b>	<b>Days Tested at 100 psi</b>	<b>Permeability (mD)</b>
1	1	7.36
2	1	8.63
3	1	2.29
4	25	1.27
5	30	0
6	18	0.38
7	1	5.97
8	1	32.12
9	1	50.53
10	1	35.29



Table 21 summarizes all three sets of permeability models.

**Table 21---Flows for all sets of 8-ft Permeability Models**

<b>Composition</b>	<b>Density (lb/gal)</b>	<b>Permeability (mD)</b>	<b>Days Tested at 100 psi</b>	<b>Set Number</b>
Type I + 2% SMS	13.4	7.36	1	3
Type I + 2% SMS	13.0	8.63	1	3
Type I + 2.5% SMS	13.0	0	90	2
Type I + 3% SMS	12.5	2.29	3	2
Type I + 3% SMS	12.0	3.75	1	1
TXI LW Neat	13.0	0	90	2
TXI LW + 2% SMS	12.0	0	90	2
TXI LW + 3% SMS	11.5	1.27	25	3
TXI LW + 3% SMS	11.0	2.29	1	3
65:35 Type I:Poz + 6% Gel	13.5	0	30	3
65:35 Type I:Poz + 10% Gel	13.0	0.38	25	2
65:35 Type I:Poz + 12% Gel	12.5	0.89	1	2
65:35 Type I:Poz + 16% Gel	12.0	6.73	1	2
50:50 Type I:Poz + 6% Gel	13.4	0.38	18	3
50:50 Type I:Poz + 8% Gel	12.8	5.97	1	3
50:50 Type I:Poz + 10% Gel	12.4	32.12	1	3
H + 8% Gel	12.5	35.29	1	3
H + 12% Gel	12.0	50.53	1	3
Type I + 16% Gel	13.0	0	90	2
Type I + 18% Gel	12.5	1.23	3	2
Type I + 20% Gel	12.0	2.41	1	2
Type I Neat	15.6	0	44	1
Type I + 13.2% Beads	12.0	0	44	1
Type I + 1 gal/sk Latex	15.6	0	44	1





## **Appendix A—Test Procedures**

### ***Sample Preparation***

Some preparation and testing methods were modified to adapt for the lightweight bead and foamed slurries. The mixing procedures for the bead slurry were also modified to minimize bead breakage due to high shear from API blending procedures. The following blending procedure was used for the bead slurry.

1. Weigh out the appropriate amounts of the cement, water, and beads into separate containers.
2. Mix the cement slurry (without beads) according to Section 5.3.5 of API RP 10B.
3. Pour the slurry into a metal mixing bowl and slowly add beads while continuously mixing by hand with a spatula. Mix thoroughly.
4. Pour this slurry back into the Waring blender and mix at 4,000 rev/min for 35 seconds to mix and evenly distribute the contents.

Testing methods for the foamed slurries were also modified. For example, thickening time is performed on unfoamed slurries only. Because the air in the foam does not affect the hydration rate, the slurry is prepared as usual per API RP 10B and then the foaming surfactants are mixed into the slurry by hand without foaming the slurry.

### ***Sample Curing***

Test specimens for rock properties testing are mixed in a Waring blender and poured into cylinder molds. Samples are cured for 7 days in a 45°F atmospheric water bath.

Performance test fixture molds are filled with cement mixed in the same manner. These fixtures are also cured in a 45°F water bath for 7 days prior to testing.

### ***Thickening Time Test***

Following the procedures set forth in API RP 10B<sup>1</sup>, thickening-time tests were performed on the three cement systems. The test conditions started at 80°F and 600 psi, and were ramped to 65°F and 5,300 psi in 48 minutes.

### ***Free-Fluid Test***

The free-fluid testing that was performed on the Type 1, foamed cement and bead cement came from API RP 10B. The free-fluid procedure, also referred to as operating free water procedure, uses a graduated cylinder that is oriented vertically. The slurry is maintained at 65°F, and the free fluid that accumulates at the top of the slurry is measured. See Table A1 for test results.



**Table A1—Free Fluid Test Results**

<b>Slurry System</b>	<b>Thickening Time to 100 Bc (hr:min)</b>	<b>Percentage of Free Fluid</b>
Neat	4:38	0.8
Foamed	3:42	0.0
Bead	5:04	0.8

### ***Compressive Strength***

The compressive strengths were derived using the 2-in. cube crush method specified in API RP 10B. The samples were cured in an atmospheric water bath at 45°F. The reported values were taken from the average of three samples.

### ***Young's Modulus and Poisson's Ratio Testing***

Traditional Young's modulus testing was performed using ASTM C469<sup>2</sup>, Standard Test Method for Static Modulus of Elasticity (Young's Modulus) and Poisson's Ratio of Concrete in Compression.

The following procedure is used for the Young's modulus testing.

1. Each sample is inspected for cracks and defects.
2. The sample is cut to a length of 3.0 in.
3. The sample's end surfaces are then ground to get a flat, polished surface with perpendicular ends.
4. The sample's physical dimensions (length, diameter, weight) are measured.
5. The sample is placed in a Viton jacket.
6. The sample is mounted in the Young's modulus testing apparatus.
7. The sample is brought to 100-psi confining pressure and axial pressure. The sample is allowed to stand for 15 to 30 min until stress and strain are at equilibrium. (In case of an unconfined test, only axial load is applied.)
8. The axial and confining stress are then increased at a rate of 25 to 50 psi/min to bring the sample to the desired confining stress condition. The sample is allowed to stand until stress and strain reach equilibrium.
9. The sample is subjected to a constant strain rate of 2.5 mm/hr.
10. During the test, the pore-lines on the end-cups of the piston are open to atmosphere to prevent pore-pressure buildup.

After the sample fails, the system is brought back to the atmospheric stress condition. The sample is removed from the cell and stored.

### ***Hydrostatic Cycling and Anelastic Strain***

Hydrostatic cycling testing was then performed on cement specimens in the same load configuration as for Young's modulus and Poisson's ratio. This testing was conducted with axial loading and radial loading being maintained equally throughout the load ramping process. For such testing, the hydrostatic pressure is cycled through the



following ramping procedures.

1. Ramp up to 1,000 psi.
2. Ramp down to 100 psi.
3. Ramp up to 1,500 psi.
4. Ramp down to 100 psi.
5. Ramp up to 2,000 psi.
6. Ramp down to 100 psi.
7. Continue to failure.

Each ramp was conducted at 16.7 psi/min and the sample was held at the destination hydrostatic pressures (i.e., 100; 1,000; 1,500; and 2,000 psi) for no longer than two minutes before proceeding to the next ramp step.

Hydrostatic cycling was studied further to investigate the deformation that occurs during each of the ramps. The value (size) of the sample at 250 psi during the first ramp to 1,000 psi is the reference value for determining the percentile of deformation. This reference value (sample size) is then compared to the sample size at 250 psi during each subsequent ramp step.

Concern over the ability to compare results of this testing among different compositions led to the development of a test for determining strain and cyclic loading effects under similar conditions with respect to each composition's ultimate strength. This test is referred to as anelastic strain testing.

Anelastic strain testing, a variation of hydrostatic testing, is designed to allow a more accurate evaluation of permanent strain resulting from stressing different test compositions. Samples are cycled to 25%, 50%, and 75% of each composition's compressive strength under 500-psi confining stress. Measurement of anelastic strain with cycling provides a more comparable value of each composition's performance. The first step in the procedure involves compression testing a sample to failure in the load cell with 500-psi confining stress. Once this failure load value is determined, additional samples will be tested by applying axial loads equal to 25%, 50%, and 75% of the failure load, and cycling until samples fail. The cyclic loading rate will be maintained at 250 psi/min and the confining force will be maintained at 500 psi. Plastic deformation will be measured at the end of each cycle. Results will include cycles to failure and anelastic strain per cycle. CT scans will be performed on each sample prior to testing to rule out the presence of any large voids.

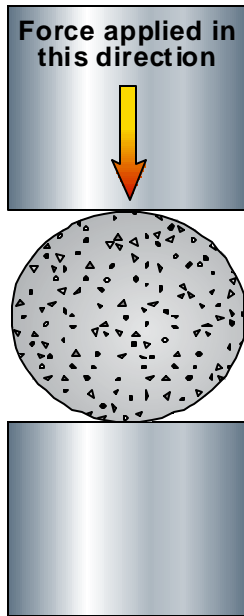
### ***Tensile Strength and Tensile Young's Modulus***

Tensile strength was tested using ASTM C496<sup>3</sup> (Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens). For this testing, the specimen dimensions were 1.5 in. diameter by 1 in. long. **Figure A1** shows a general schematic of how each specimen is oriented on its side during testing. The force was applied by constant displacement of the bottom plate at a rate of 1 mm every 10 minutes. Change in the specimen diameter can be calculated from the test plate displacement. The



(compressive) strength of the specimen during the test can be graphed along with the diametric strain (change in diameter/original diameter) to generate the tensile Young's modulus. Strain was measured by a linear displacement transducer mounted to record diameter continuously as stress was applied.

**Figure A1—Sample Orientation for ASTM C496-90 Testing**



### ***Annular Seal Testing Procedure***

Samples for annular seal testing are prepared by mixing cement compositions, pouring them into specified molds, and curing them for 7 days in 80°F water baths. After curing, three specimens for each test composition and condition are tested.

These procedures are for use with the annular seal apparatus. Specific procedures are applied as necessary for each formation simulation: soft, intermediate, and hard. The soft apparatus test procedure is to be used with cores cured to set in a soft gel mold, which provides a semi-restricting force on the outside of the core. The intermediate specimen mold uses a 3-in. diameter Schedule 40 PVC pipe as the outer containment. The hard apparatus uses a 3-in. Schedule 40 steel pipe as the outside containment, giving the cement slurry a restricting force outside of the core. The hard-formation configuration consists of a sandblasted internal pipe with an outer diameter (OD) of  $1 \frac{1}{16}$  in. and a sandblasted external pipe with an internal diameter (ID) of 3 in. Both pipes are 6 in. long. A contoured base and top are used to center the internal pipe within the external pipe. The base extends into the annulus 1 in. and cement fills the annulus to a height of 4 in. The top inch of annulus contains water.

For the soft-formation annular seal tests, plastisol is used to allow the cement to cure in a



less-rigid, lower-restraint environment. Plastisol is a mixture of a resin and a plasticizer that creates a soft, flexible substance. This particular plastisol blend (PolyOne's Denflex PX-10510-A) creates a substance with a hardness of 40 duro.

The soft formation configuration contains a sandblasted external pipe with an ID of 4 in. A molded plastisol sleeve with an ID of 3.0 in. and uniform thickness of 0.5 in. fits inside the external pipe. With the aid of a contoured base and top, a sandblasted internal pipe with an OD of  $1 \frac{1}{16}$  in. is then centered within the plastisol sleeve. The pipes and sleeve are 6 in. long. The base extends into the annulus 1 in. and cement fills the annulus to a height of 4 in. between the plastisol sleeve and the inner  $1 \frac{1}{16}$  -in. pipe. The top inch of annulus is filled with water.

The intermediate formation test fixture features the same configuration as the hard formation fixture except the outer pipe is made of PVC.

### **Soft-Formation Simulation**

1. After the core is cured, place the core inside the gel mold sleeve.
2. Place the core and sleeve inside the pipe-in-soft steel cell.
3. Once inside, both ends of the core are supported with O-rings.
4. The O-rings are then tightened by interior end plates to close off leaks that might be present.
5. Using water, pressurize the exterior circumference of the sleeve to 25 psi and check for leaks on the ends of the cell.
6. Cap off both ends of the steel cell with the cell end caps. One end cap has a fitting that allows for N<sub>2</sub> gas to be applied into the cell, and the other end cap allows gas to exit the cell.
7. Attach the pressure inlet line to the bottom of the cell and attach the pressure outlet line to the top of the cell.
8. Apply pressure to the inlet line (do not exceed 20 psig) and measure the flow out using flow meters.

### **Hard-Formation Simulation**

1. After the core is cured inside the steel pipe, cap off each end of the pipe with steel end caps. Each end cap has a fitting that allows for gas to be applied into the pipe or to exit the pipe.
2. Attach the pressure inlet line to the bottom of the pipe, and attach the pressure outlet line to the top of the pipe.
3. Apply pressure to the inlet line (do not exceed 20 psig) and measure the pressure out of the outlet line using flow meters.

### **Intermediate Formation Simulation**

The test fixture for performing tests with a simulated intermediate formation is very similar to that used for tests with simulated hard formations, except the outer pipe is made of Schedule 40 PVC. Stress is applied to the specimens by applying hydraulic



pressure or heat to the inner pipe.

Thermal cycling resulted from the insertion of heaters into the inner pipe and the heating of the inner pipe from 80° to 180°F over an 8 hour period then allowing the pipe to cool to 80°F. Flow through the model was measured continuously with flowmeters throughout each cycle, and cycles were repeated a minimum of five times per sample. Three specimens of each composition were tested.

To ensure that sufficient stress could be applied to induce failure in all samples, the thermal cycling test procedure was modified to allow use of a thicker-walled inner pipe that provides more steel volume for expansion. The modified test fixture now features an inside pipe with a 1.68-in. outside diameter and a 1.25-in. inside diameter, giving a wall thickness of 0.190 in. Additionally, the outer containment diameter will be increased to 3 in.

Pressure cycling resulted from the application of hydraulic pressure to the inner pipe. For the initial cycle, pressure was increased from 0 to 1000 psi. Pressure was then released and allowed to return to 0, and flow measurements were made. Additional cycles were made by increasing the upper pressure limit by 1000 psi (0 to 1,000 to 0 psi, 0 to 2,000 to 0 psi, etc.) and measuring flow at the endpoint (0) of each cycle. If specimens were cycled to 10,000 psi without failure, the 0 to 10,000 to 0 psi pressure cycle was repeated a minimum of five times. The original test procedure was modified to establish a maximum pressure of 10,000 psi during pressure cycles.

All modified testing methods performed with intermediate formations were applied to soft and hard formations also. Hard formations incorporated additional pressure cycles to 10,000 psi until achieving failure.

### ***Shear Bond Strength Testing***

Shear bond strength tests are used for investigating the effect that restraining force has on shear bond. Samples are cured in a hard-formation configuration (**Figure A2**) and in a soft-formation configuration (**Figure A3**). The hard-formation configuration consists of a sandblasted internal pipe with an outer diameter (OD) of 1 <sup>1</sup>/<sub>16</sub> in. and a sandblasted external pipe with an internal diameter (ID) of 3 in. Both pipes are 6 in. long. A contoured base and top are used to center the internal pipe within the external pipe. The base extends into the annulus 1 in. and cement fills the annulus to a height of 4 in. The top inch of annulus contains water.

For the soft-formation shear bond tests, plastisol is used to allow the cement to cure in a less-rigid, lower-restraint environment. Plastisol is a mixture of a resin and a plasticizer that creates a soft, flexible substance. This particular plastisol blend (PolyOne's Denflex PX-10510-A) creates a substance with a hardness of 40 duro.

The soft formation configuration contains a sandblasted external pipe with an ID of 4 in. A molded plastisol sleeve with an ID of 3.0 in. and uniform thickness of 0.5 in. fits inside

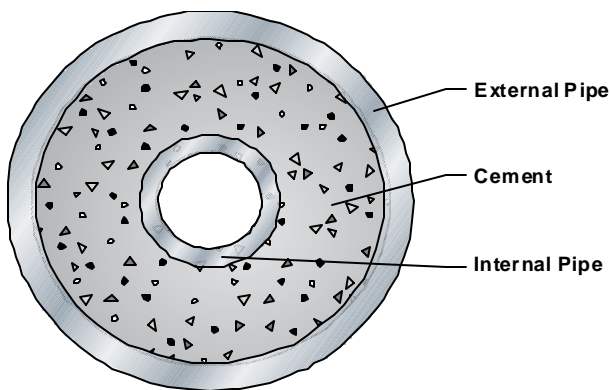


the external pipe. With the aid of a contoured base and top, a sandblasted internal pipe with an OD of  $1 \frac{1}{16}$  in. is then centered within the plastisol sleeve. The pipes and sleeve are 6 in. long. The base extends into the annulus 1 in. and cement fills the annulus to a height of 4 in. between the plastisol sleeve and the inner  $1 \frac{1}{16}$ -in. pipe. The top inch of annulus is filled with water.

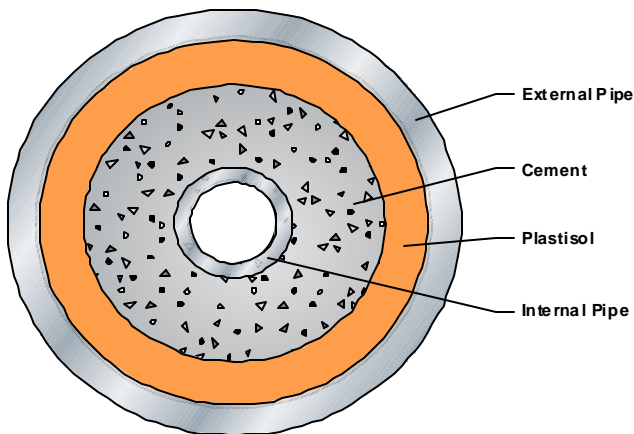
The intermediate formation test fixture features the same configuration as the hard formation fixture except the outer pipe is made of PVC.

Cycling tests for the shear bond specimens follow all cycling procedures used for testing the annular seals. Once the annular seal cycles are performed the shear bond measurements are then taken. This allows correlation with annular seal test results. Shear bonds are measured after the cycling to determine the level of bond remaining.

**Figure A2—Cross-section of pipe-in-pipe test fixture configuration for shear bond test.**



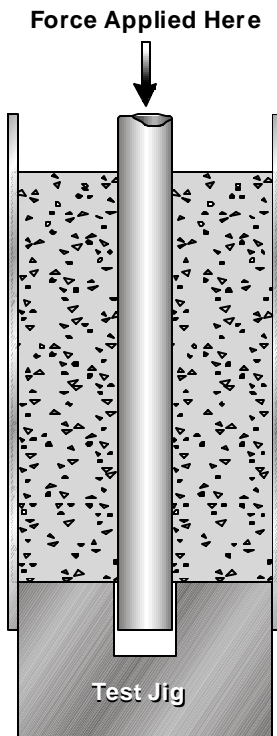
**Figure A3—Cross-section of pipe-in-soft test fixture configuration for shear bond test.**





The shear bond measures the stress necessary to break the bond between the cement and the internal pipe. This was measured with the aid of a test jig that provides a platform for the base of the cement to rest against as force is applied to the internal pipe to press it through. (Figure A4) The shear bond force is the force required to move the internal pipe. The pipe is pressed only to the point that the bond is broken; the pipe is not pushed out of the cement. The shear bond strength is the force required to break the bond (move the pipe) divided by the surface area between the internal pipe and the cement.

**Figure A4—Test jig for testing shear bond strength**



### **Cement Column Seal Tests**

Eight-foot lengths of 2-in. Schedule 40 pipe are mounted vertically and fitted at the top and bottom with end caps equipped with pressure inlet and outlet ports. The bottom of each pipe is filled with 6 in. of 20-40 sand to provide an open base for gas injection. For the first set, sets of two fixtures are each filled with one of four different cement slurries: bead, Type 1, latex, and sodium metasilicate. Samples are covered with water and cured for 7 days under 1000-psi pressure. After the samples are cured, 100 psi of pressure is applied to the bottom of each fixture and any flow through the column is monitored. For the second and third sets, ten fixtures are each filled with ten different cement slurries. Samples are covered with water and cured for 3 days under 1000-psi pressure. After the samples are cured, 100 psi of pressure is applied to the bottom of each fixture and any flow through the column is monitored.

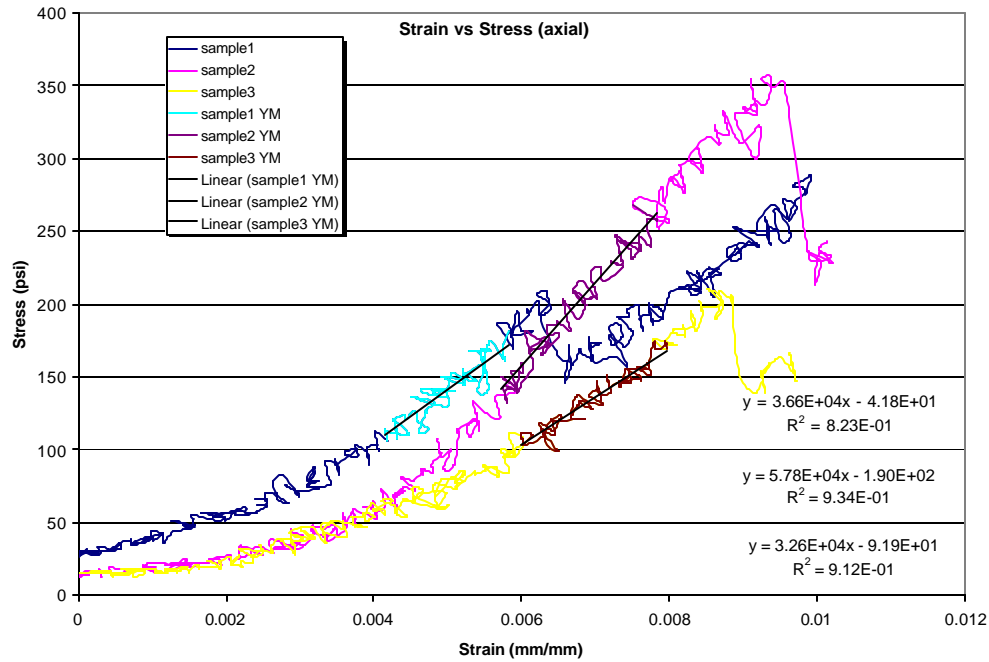




## Appendix B—Test Data

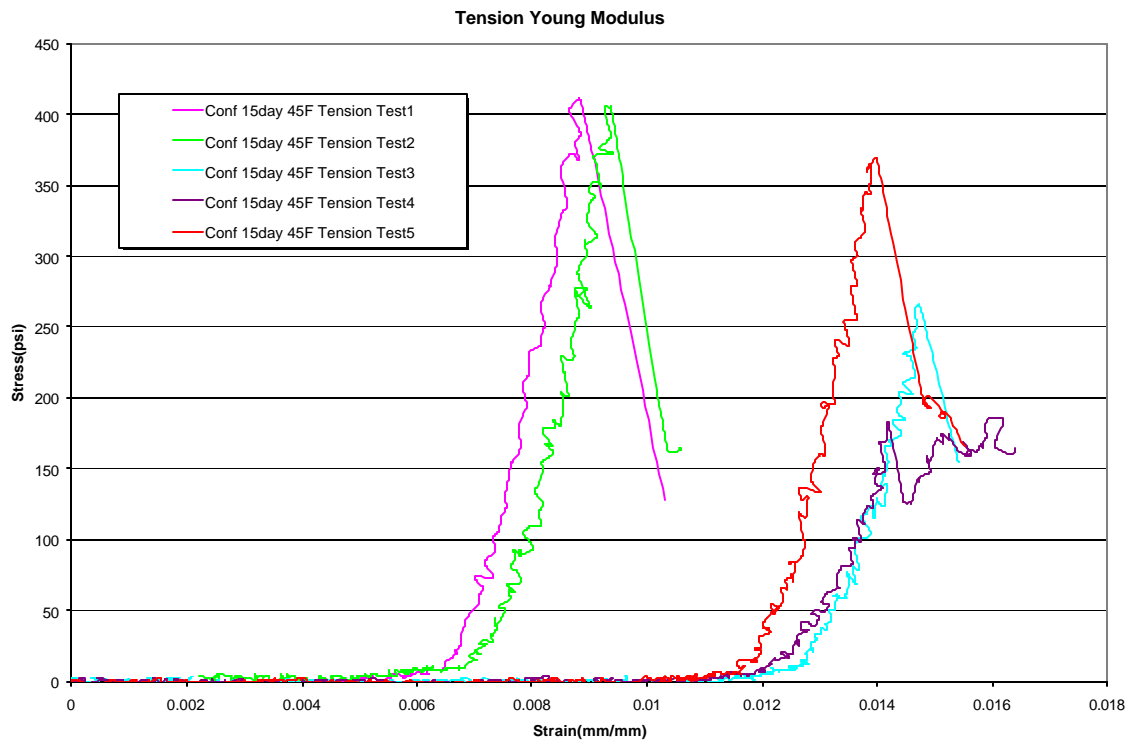
Graphical data for all mechanical properties tests performed in this investigation are presented in this appendix.

**Figure B1—Plot of tensile strength and Young’s modulus results for latex slurry with fibers (sample 1), Type 1 slurry with fibers (sample 2), and latex slurry (sample 3).**



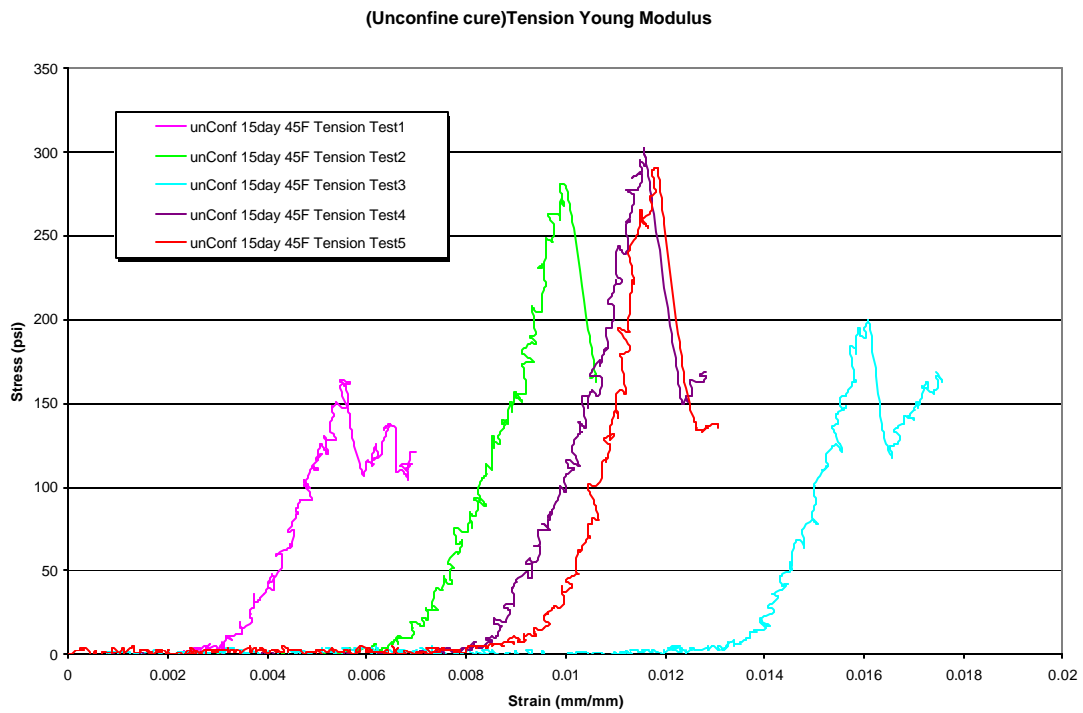


**Figure B2—Plot of tensile strength and Young's modulus results for neat Type 1 slurry cured in a confined state.**



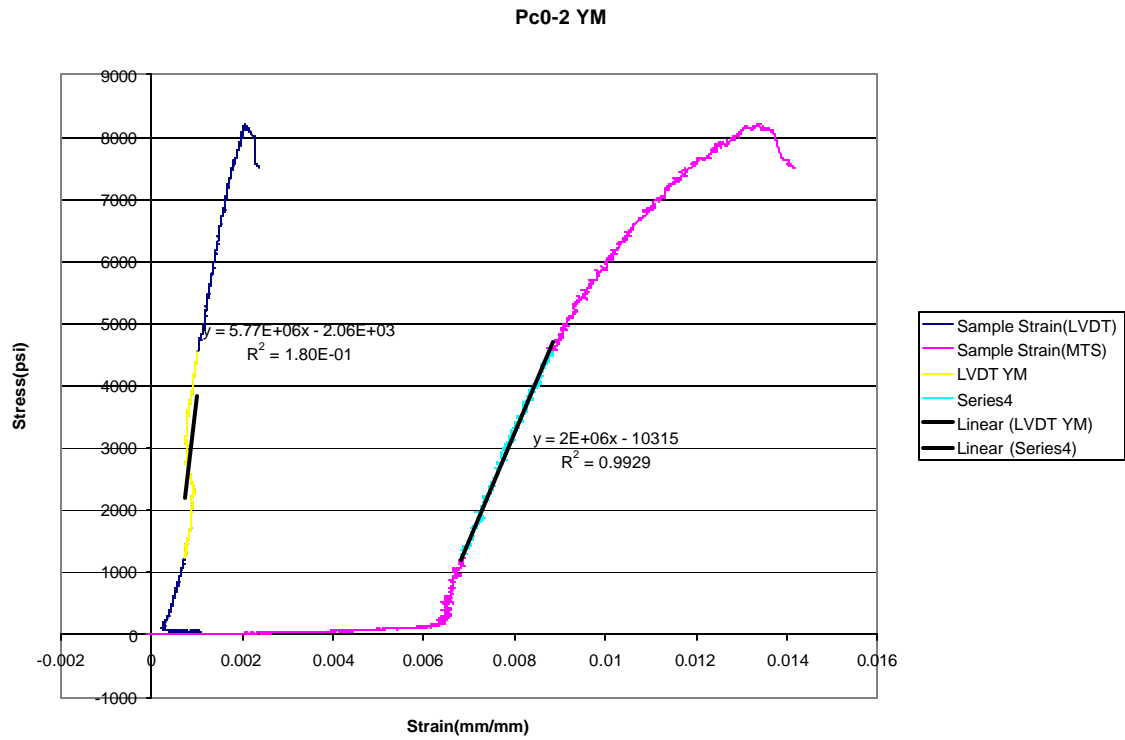


**Figure B3—Plot of tensile strength and Young’s Modulus results for 12-lb/gal foam slurry.**



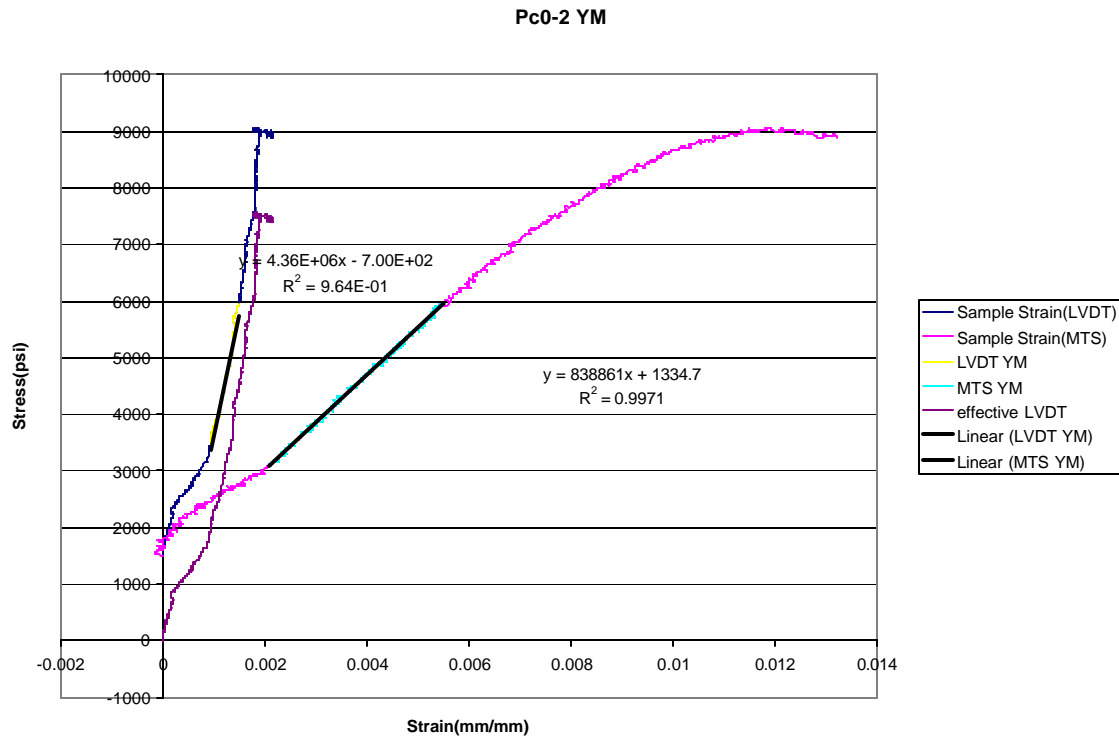


**Figure B4—Plot of compressive Young's modulus for Type 1 slurry at 0-psi confining pressure.**



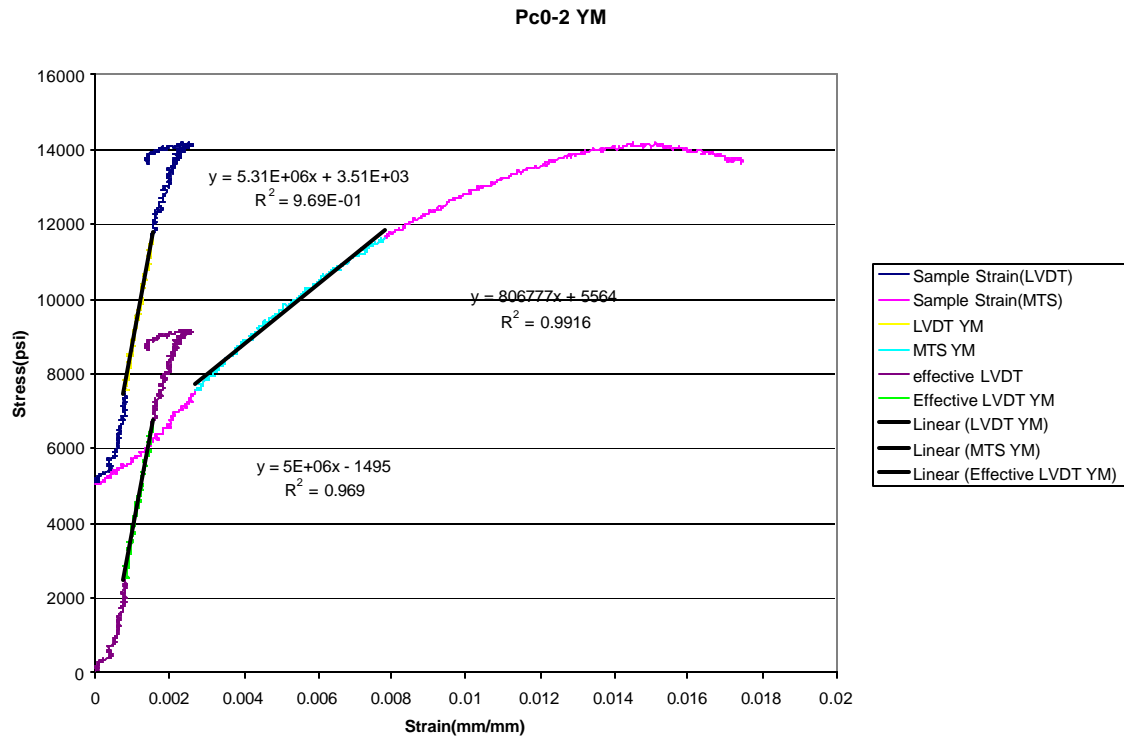


**Figure B5—Plot of compressive Young's modulus for Type 1 slurry at 1500-psi confining pressure.**



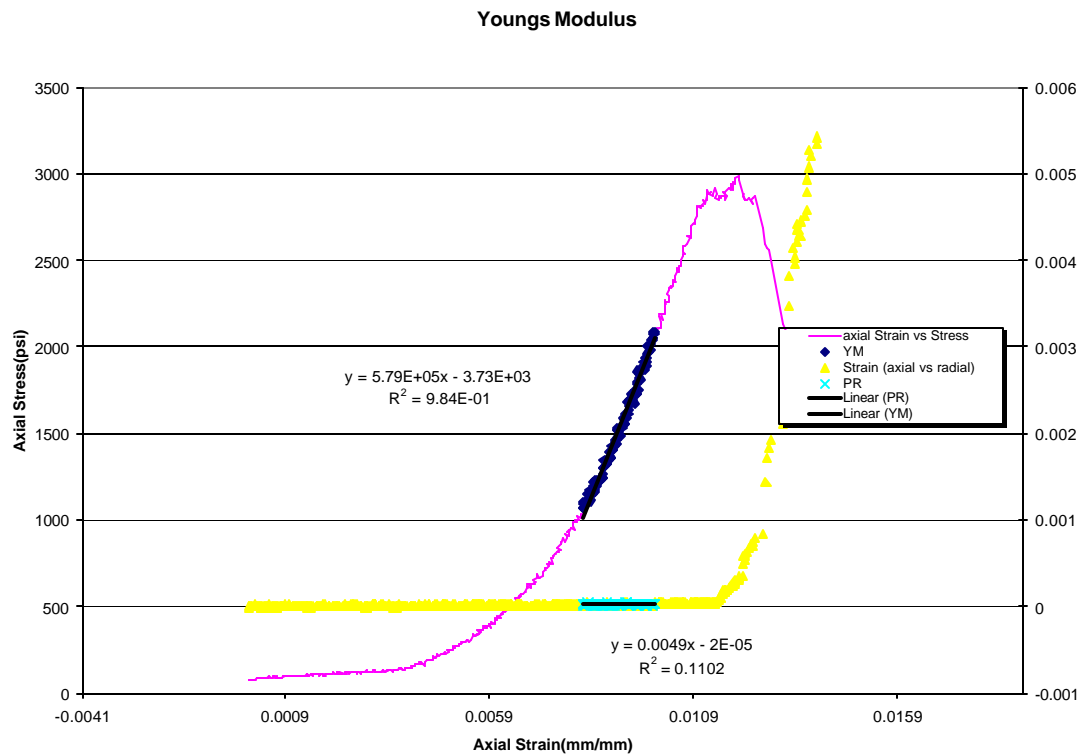


**Figure B6— Plot of compressive Young's modulus for Type 1 slurry at 5000-psi confining pressure.**



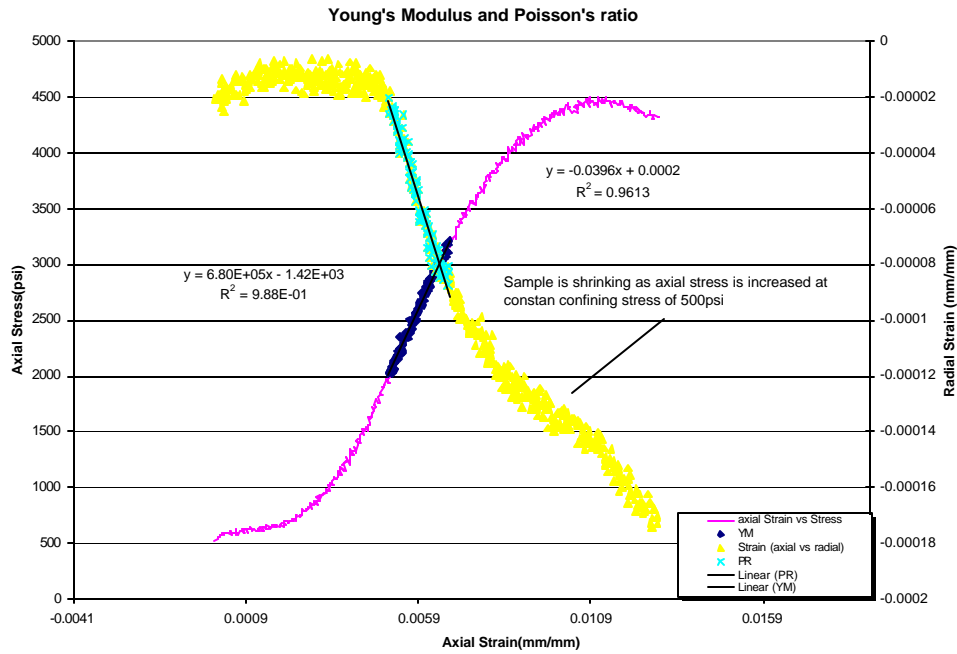


**Figure B7— Plot of compressive Young's modulus for 12-lb/gal foam slurry at 0-psi confining pressure.**

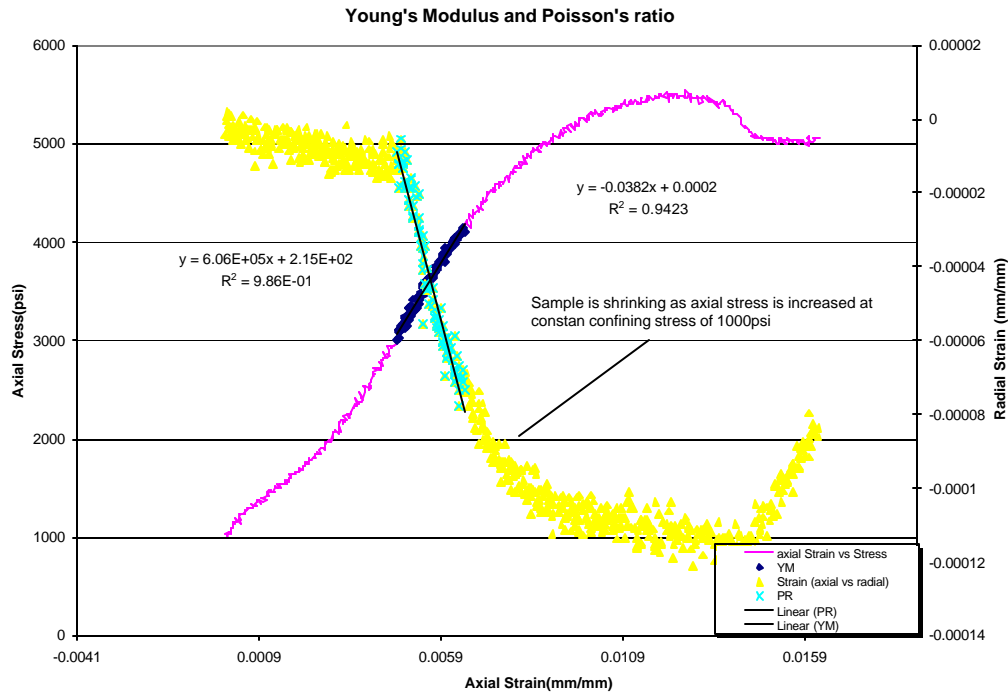




**Figure B8— Plot of compressive Young's modulus for 12-lb/gal foam slurry at 500-psi confining pressure.**



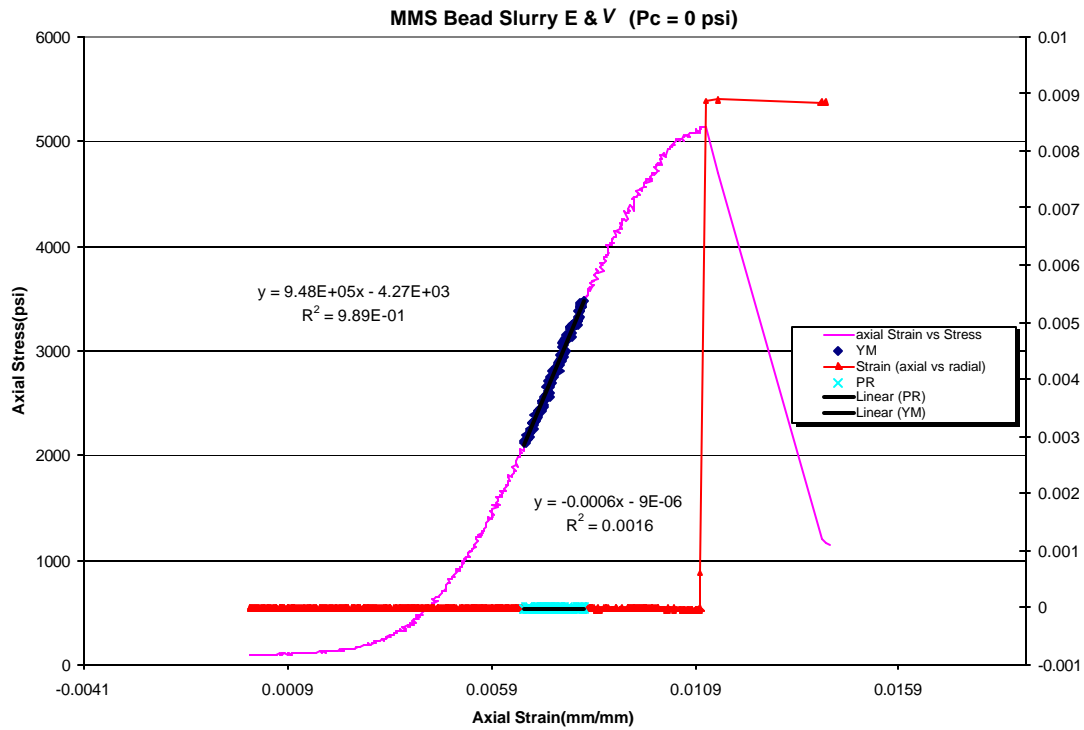
**Figure B9— Plot of compressive Young's modulus for 12-lb/gal foam slurry at 1000-psi confining pressure.**





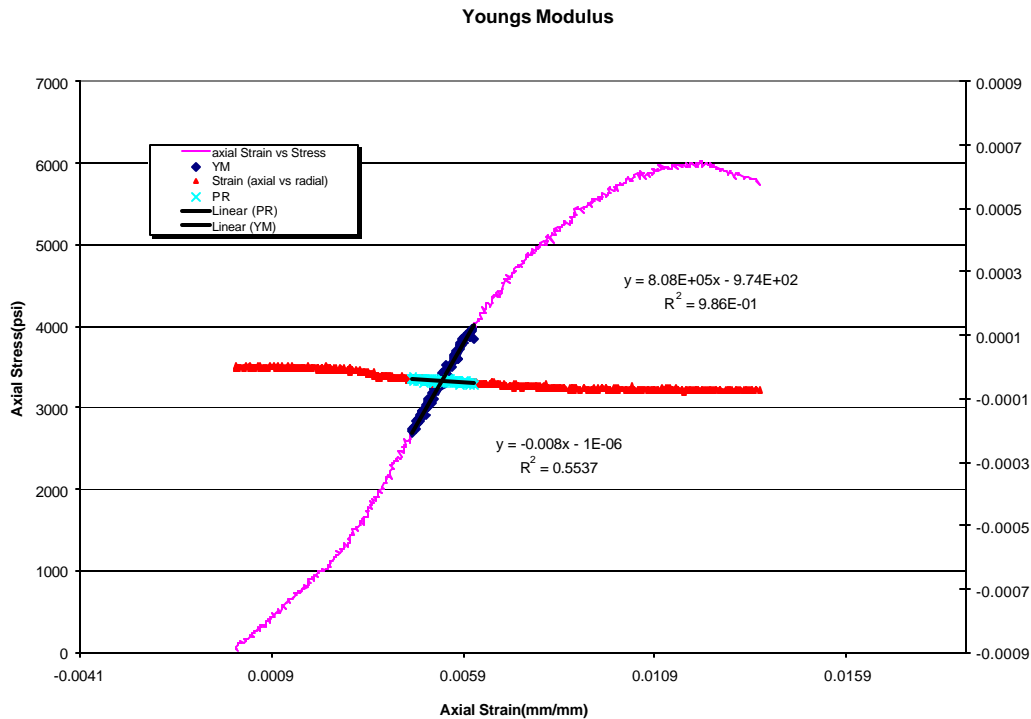


**Figure B10— Plot of compressive Young's modulus for bead slurry at 0-psi confining pressure.**





**Figure B11— Plot of compressive Young's modulus for bead slurry at 500-psi confining pressure.**



**Figure B12— Plot of compressive Young's modulus for bead slurry at 1000-psi confining pressure.**

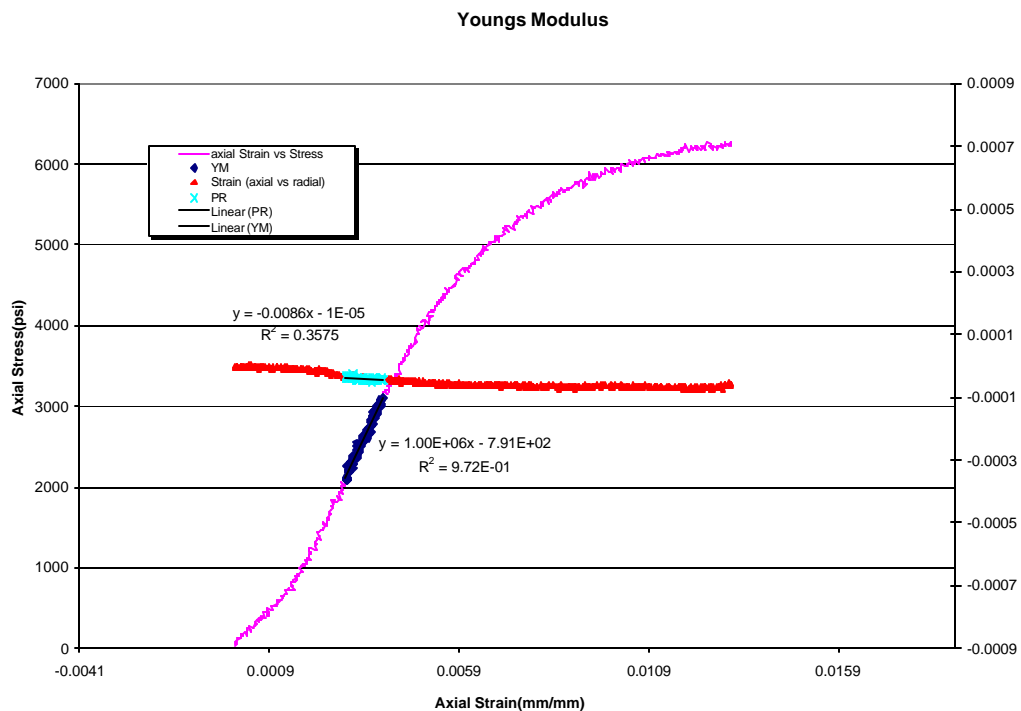




Figure B13— Plot of compressive Young's modulus for latex slurry at 0-psi confining pressure.

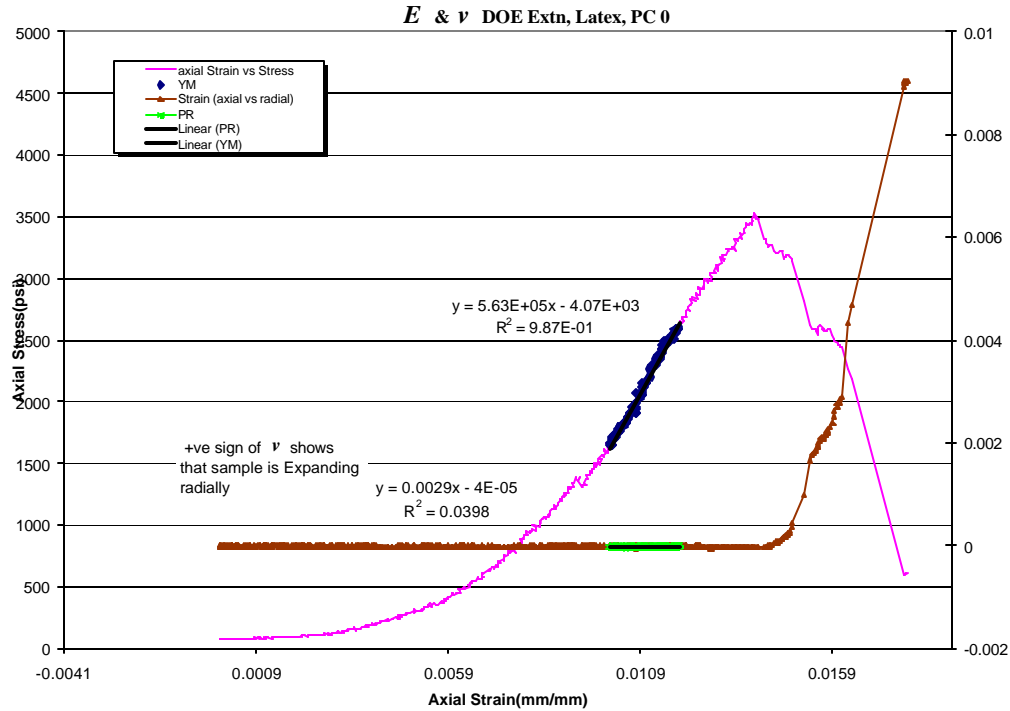
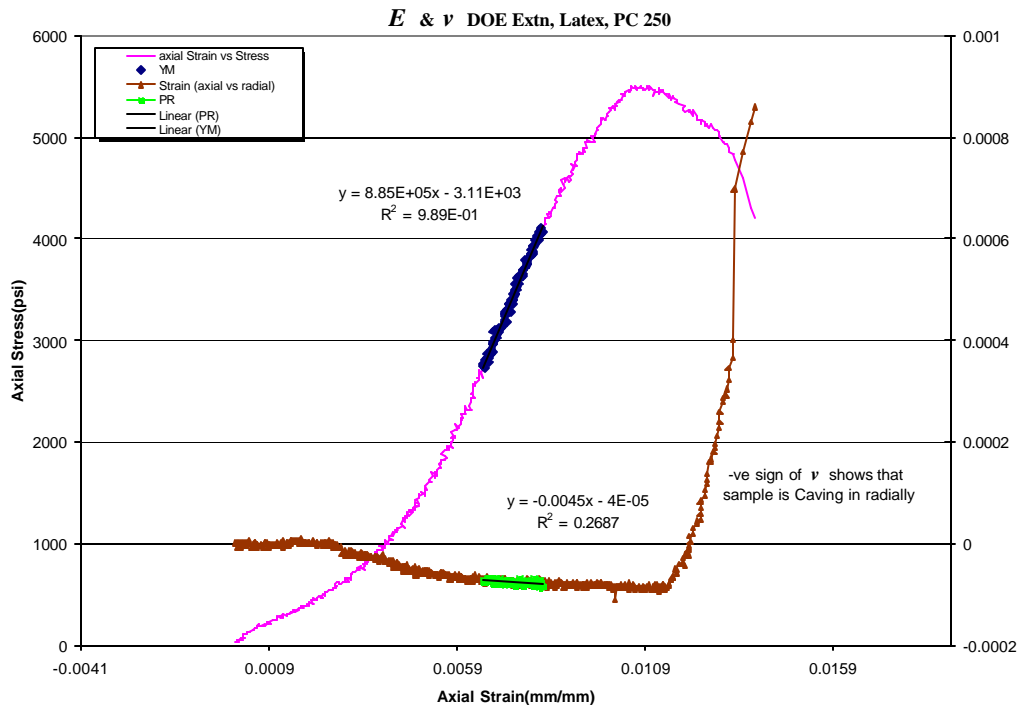
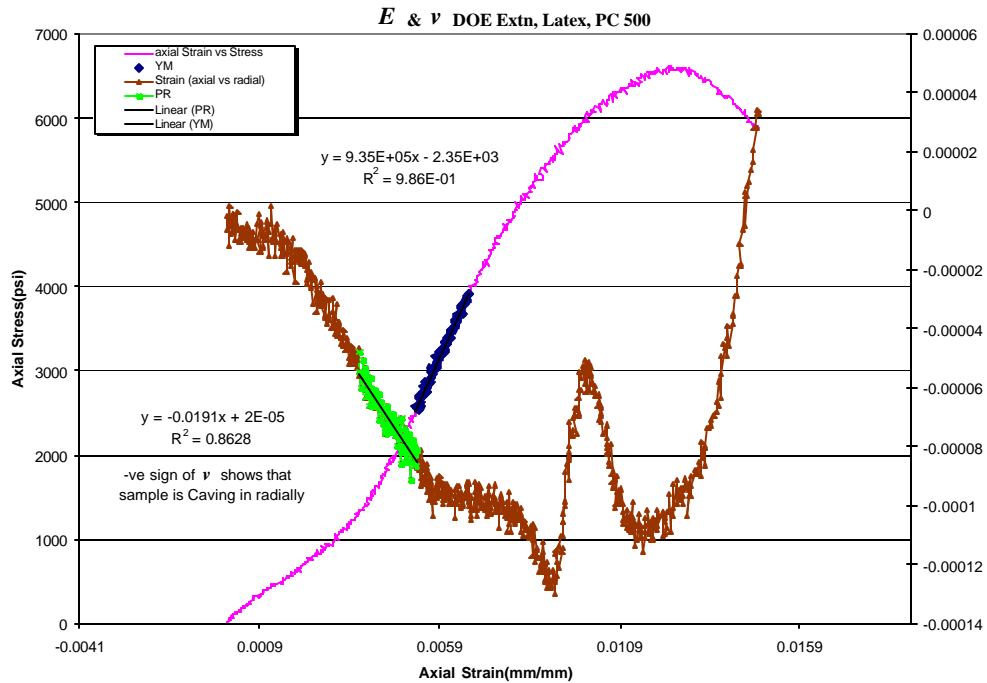


Figure B14— Plot of compressive Young's modulus for latex slurry at 250-psi confining pressure.

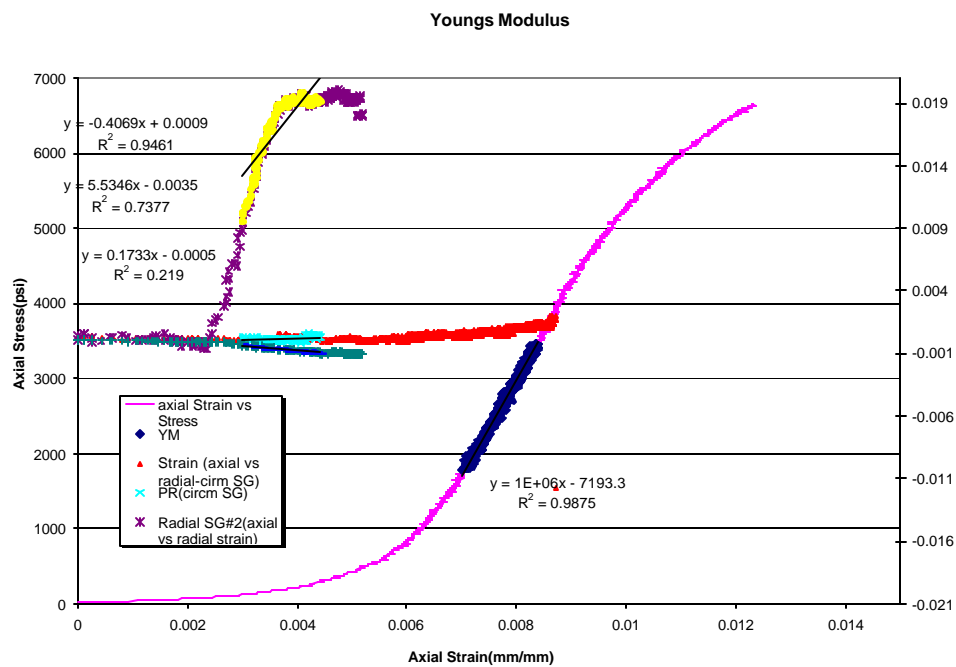




**Figure B15— Plot of compressive Young's modulus for latex slurry at 500-psi confining pressure.**

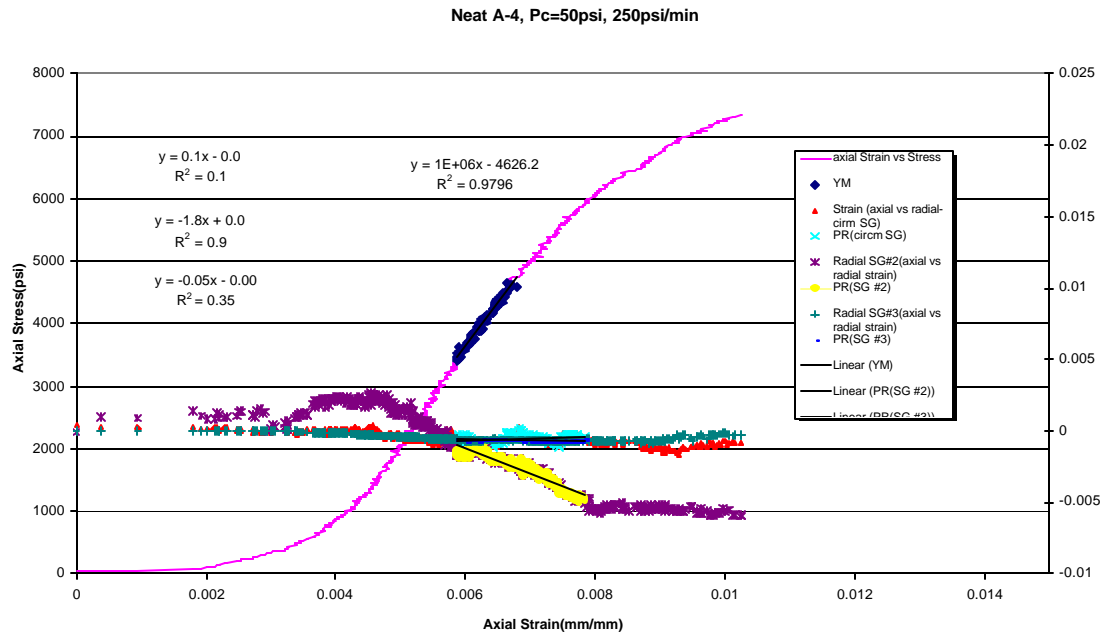


**Figure B16—Young's modulus measurements for Type 1 slurry at 500-psi confining stress and a 100-psi/min load rate.**



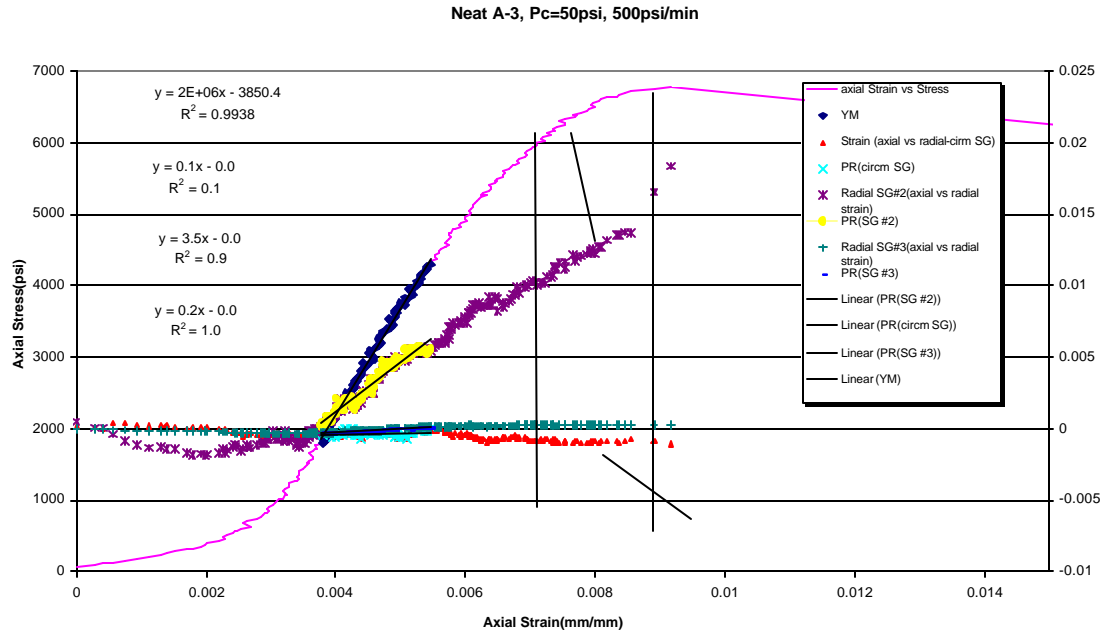


**Figure B17—Young's modulus measurements for Type 1 slurry at 500-psi confining stress and a 250-psi/min load rate.**





**Figure B18—Young's modulus measurements for Type 1 slurry at 500-psi confining stress and a 500-psi/min load rate.**



**Figure B19—Hydrostatic cycling data for bead slurry showing anelastic strain.**

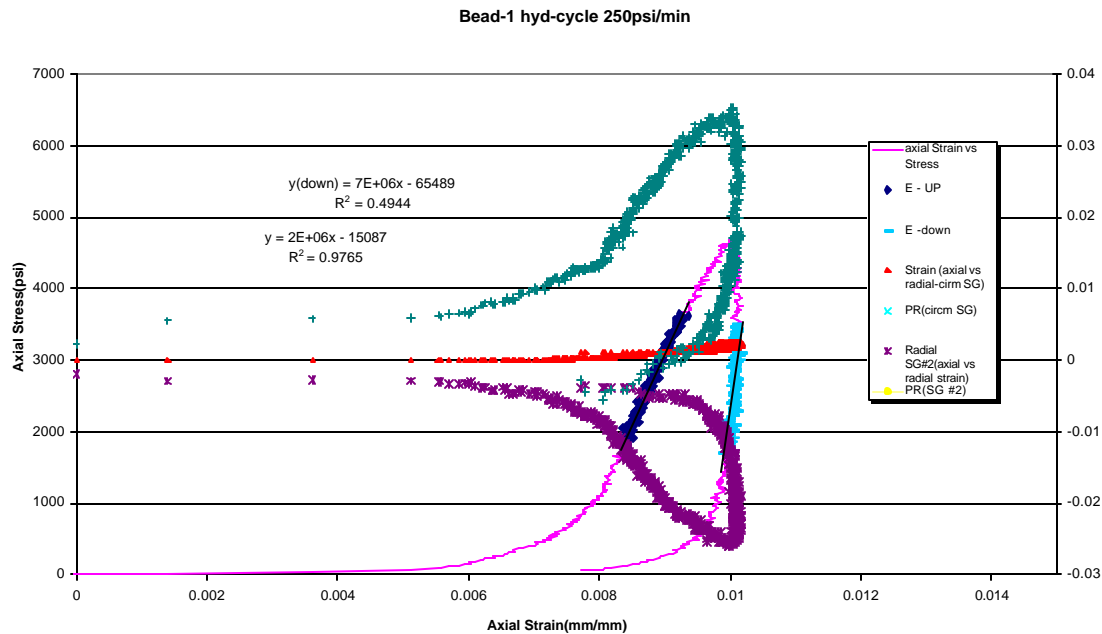




Figure B20— Hydrostatic cycling data for Class H slurry showing anelastic strain.

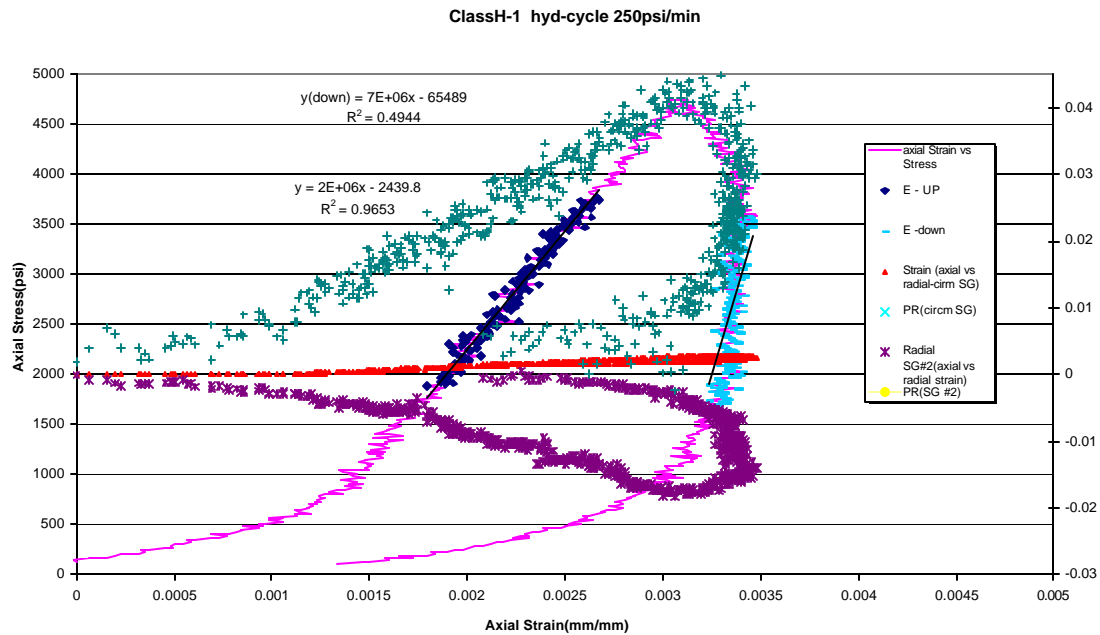


Figure B21— Hydrostatic cycling data for 12-lb/gal foam slurry showing anelastic strain.

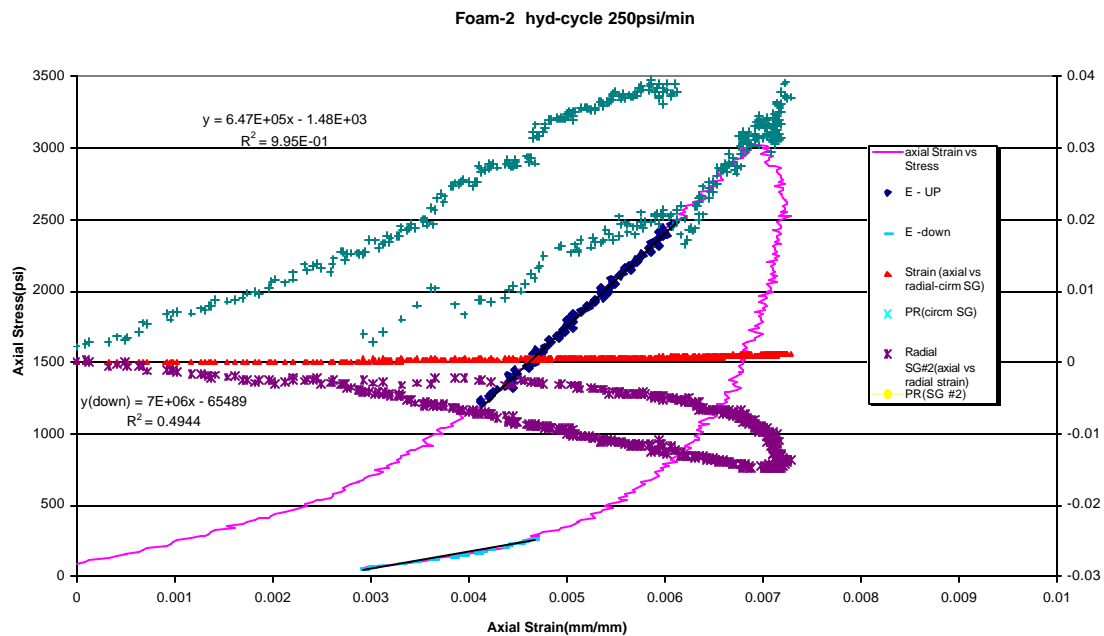




Figure B22— Hydrostatic cycling data for Type 1 slurry showing anelastic strain.

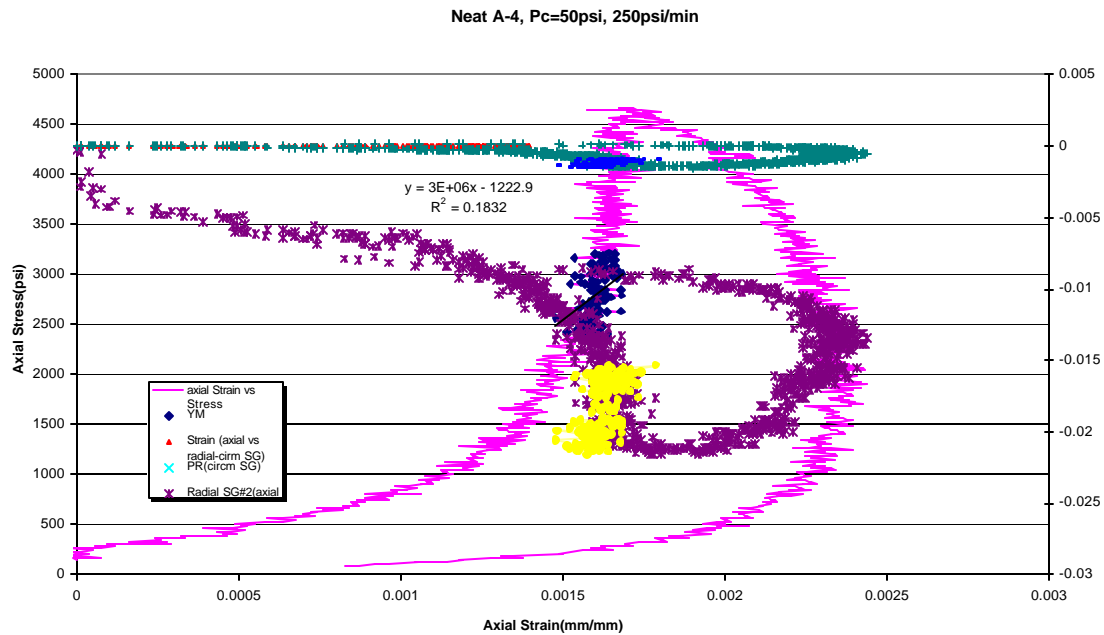
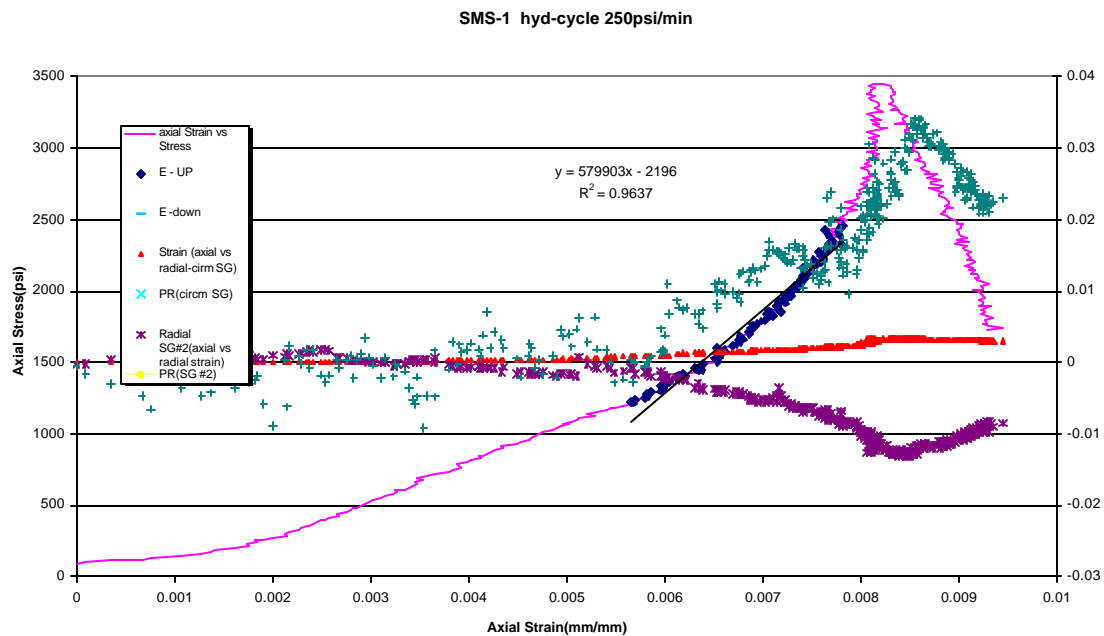


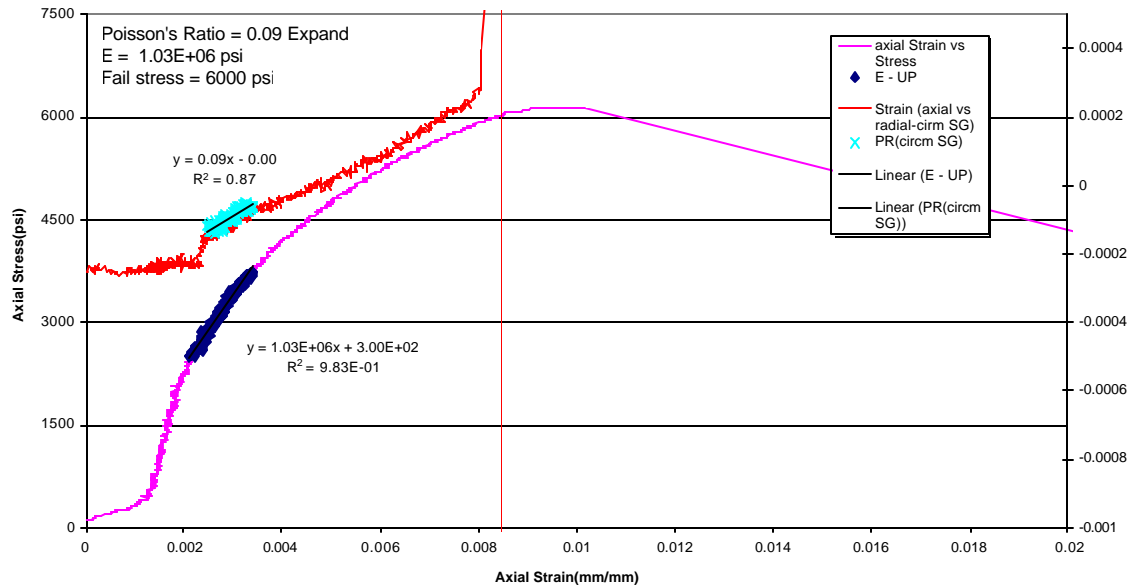
Figure B23— Hydrostatic cycling data for sodium metasilicate (SMS) slurry showing anelastic strain.



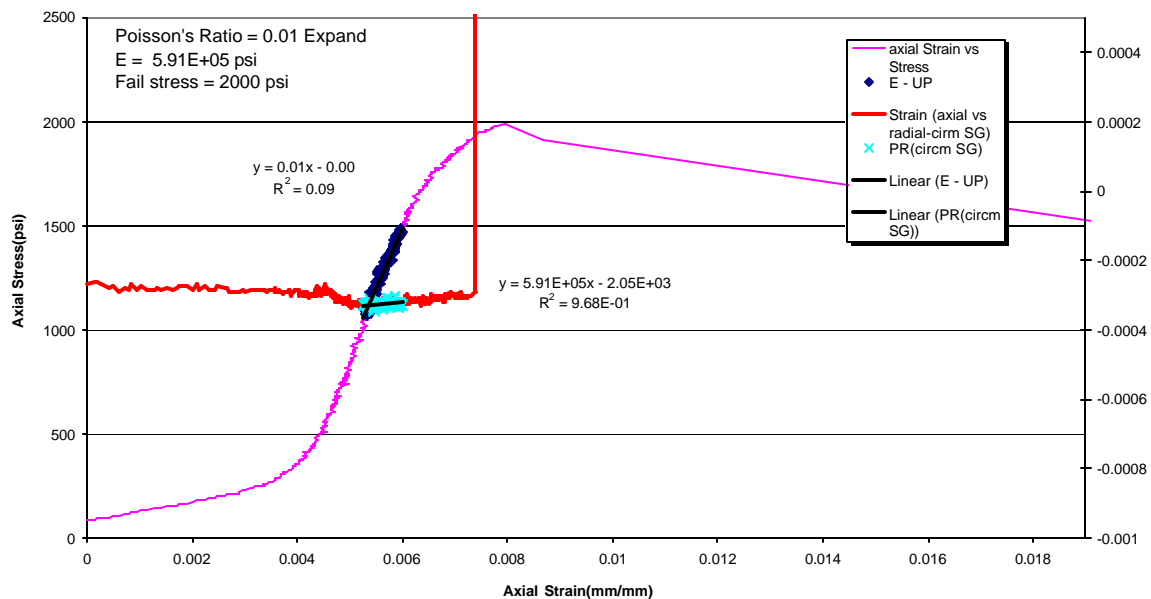




**Figure B24— Anelastic strain failure load for neat Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

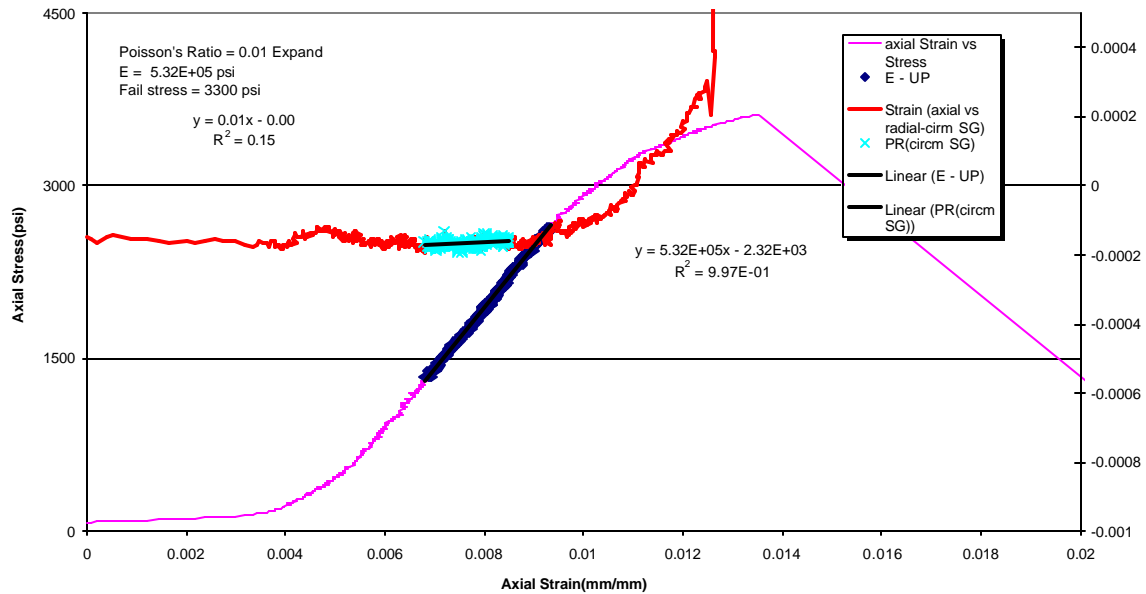


**Figure B25— Anelastic strain failure load for foam slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

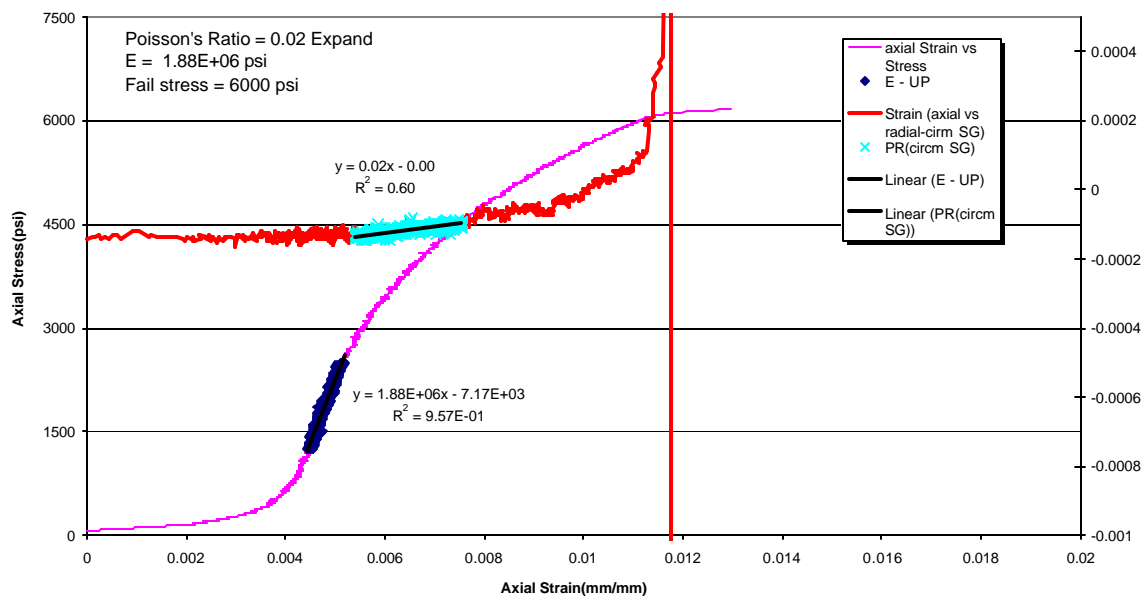




**Figure B26— Anelastic strain failure load for bead slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

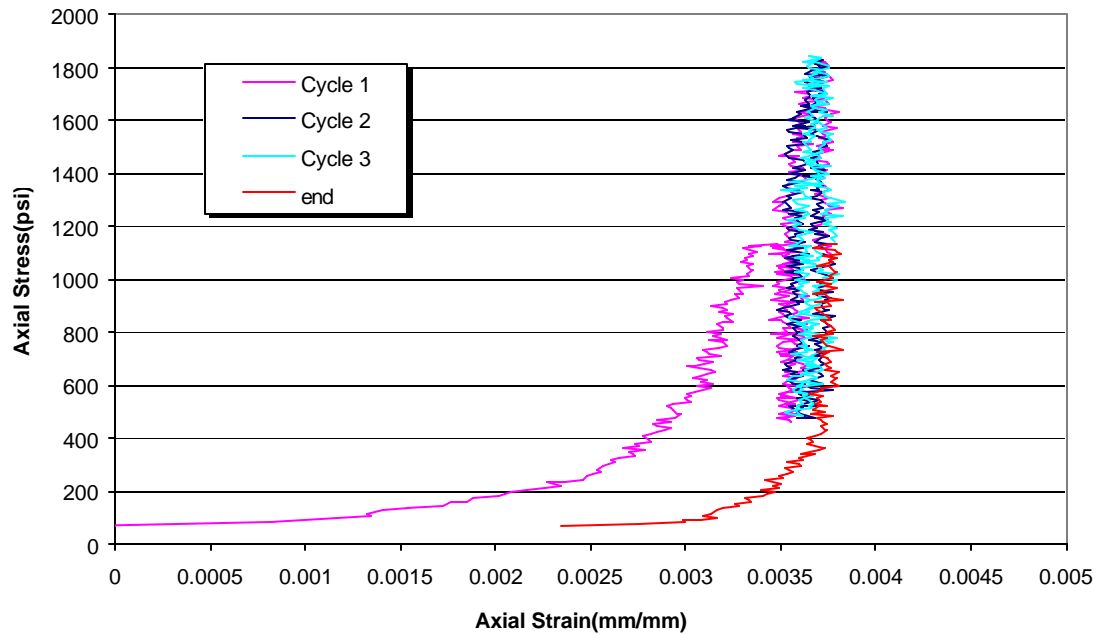


**Figure B27—Anelastic strain failure load for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

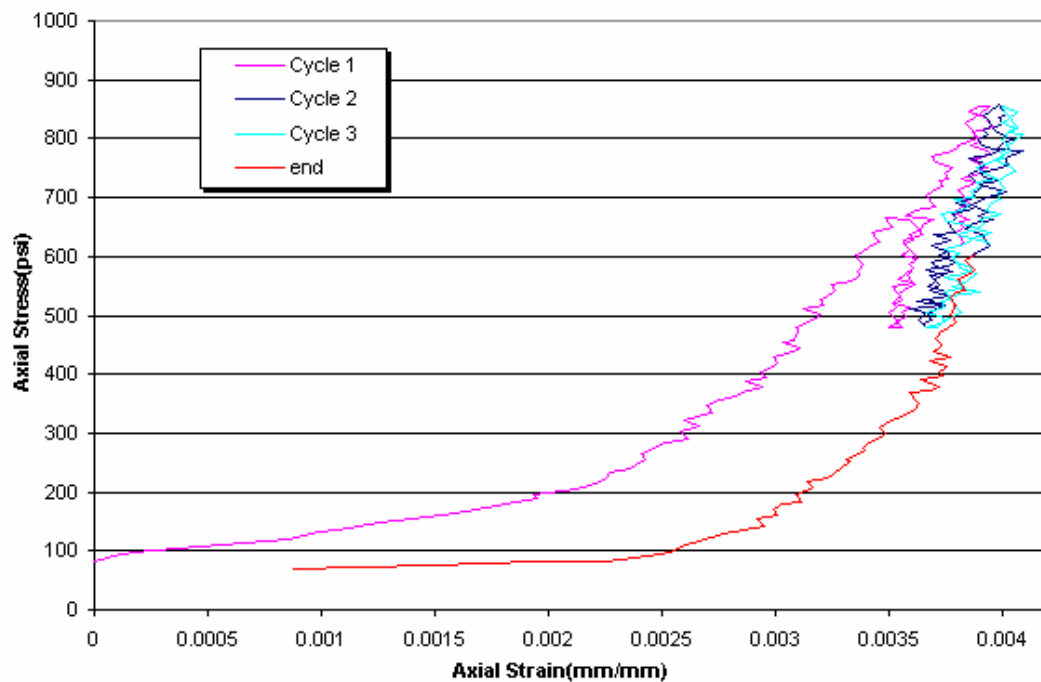




**Figure B28—Anelastic strain, cycled to 25% of failure load, for Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

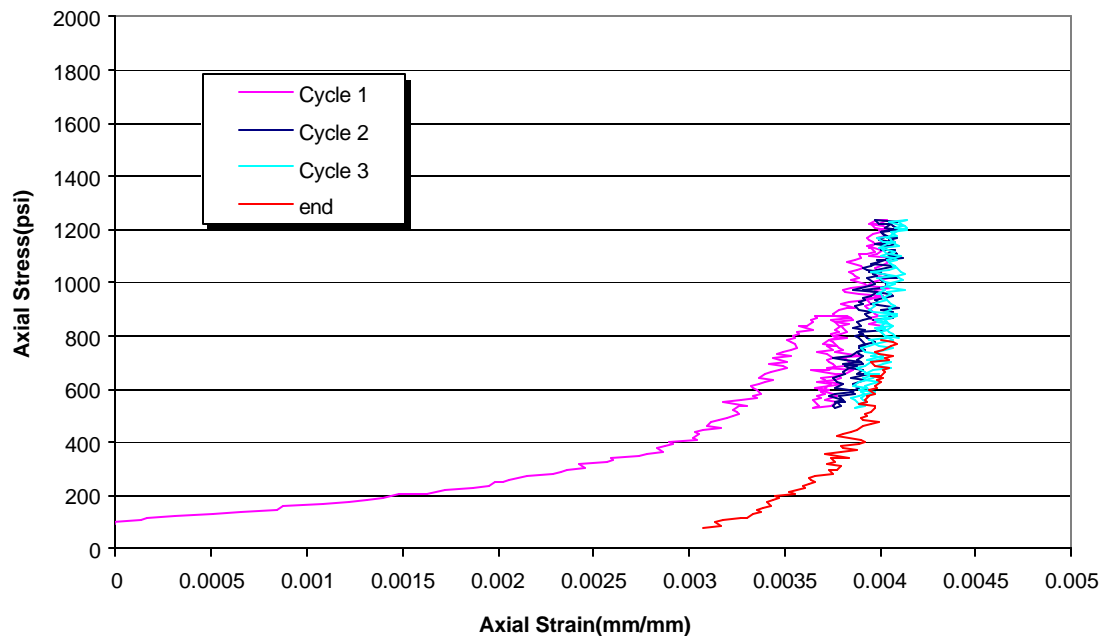


**Figure B29—Anelastic strain, cycled to 25% of failure load, for foam slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

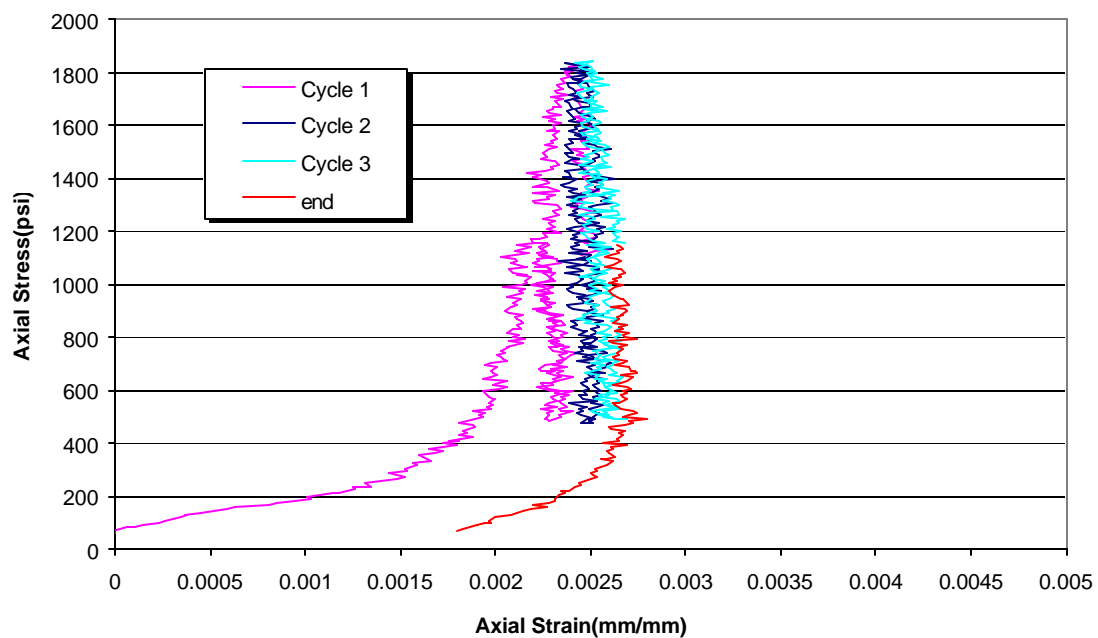




**Figure B30—Anelastic strain, cycled to 25% of failure load, for bead slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

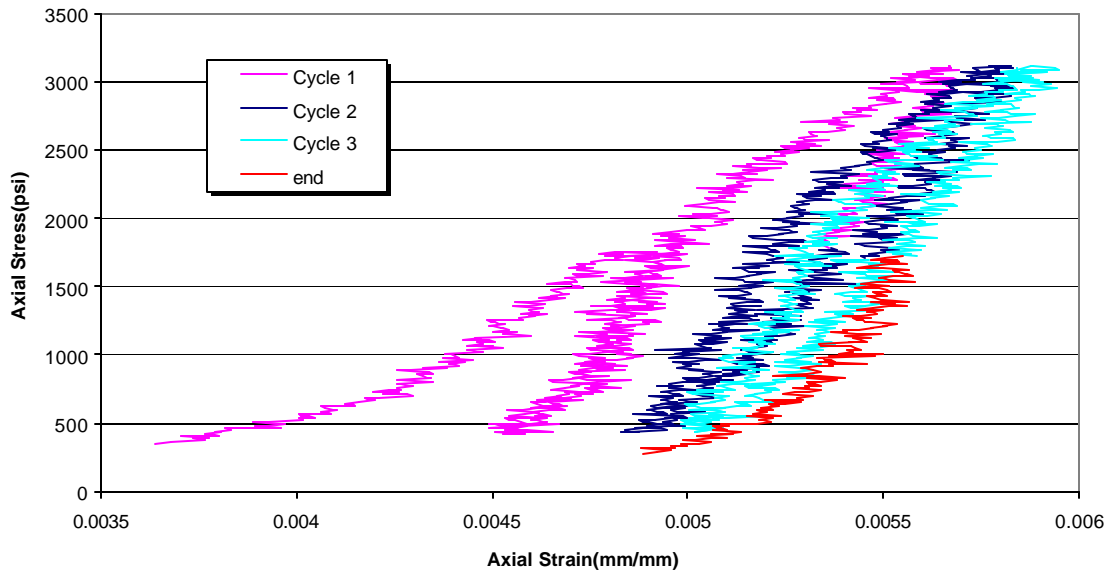


**Figure B31—Anelastic strain, cycled to 25% of failure load, for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

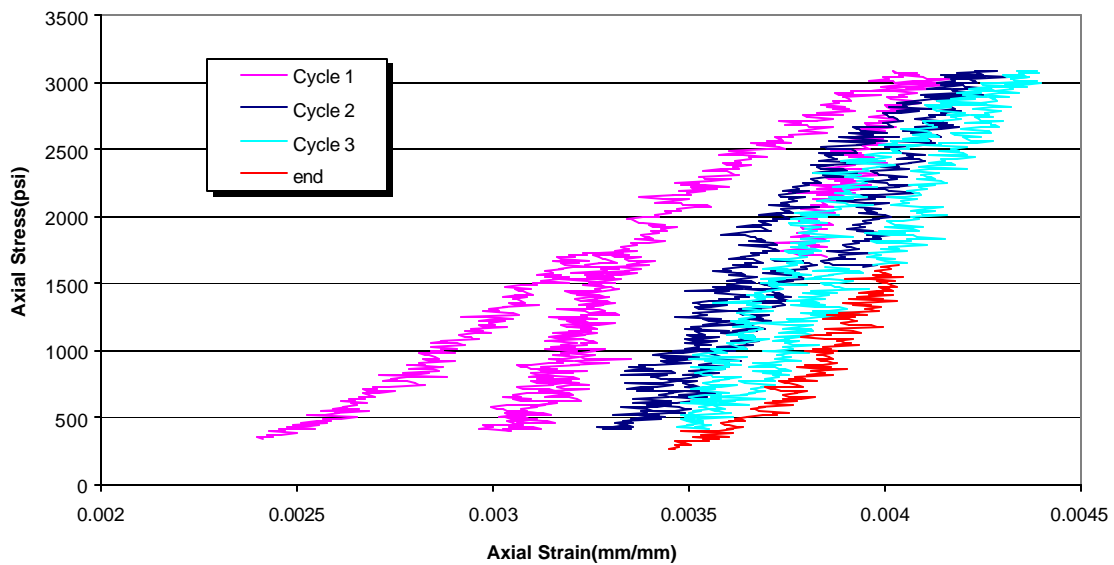




**Figure B32—Anelastic strain, cycled to 50% of failure load, for Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

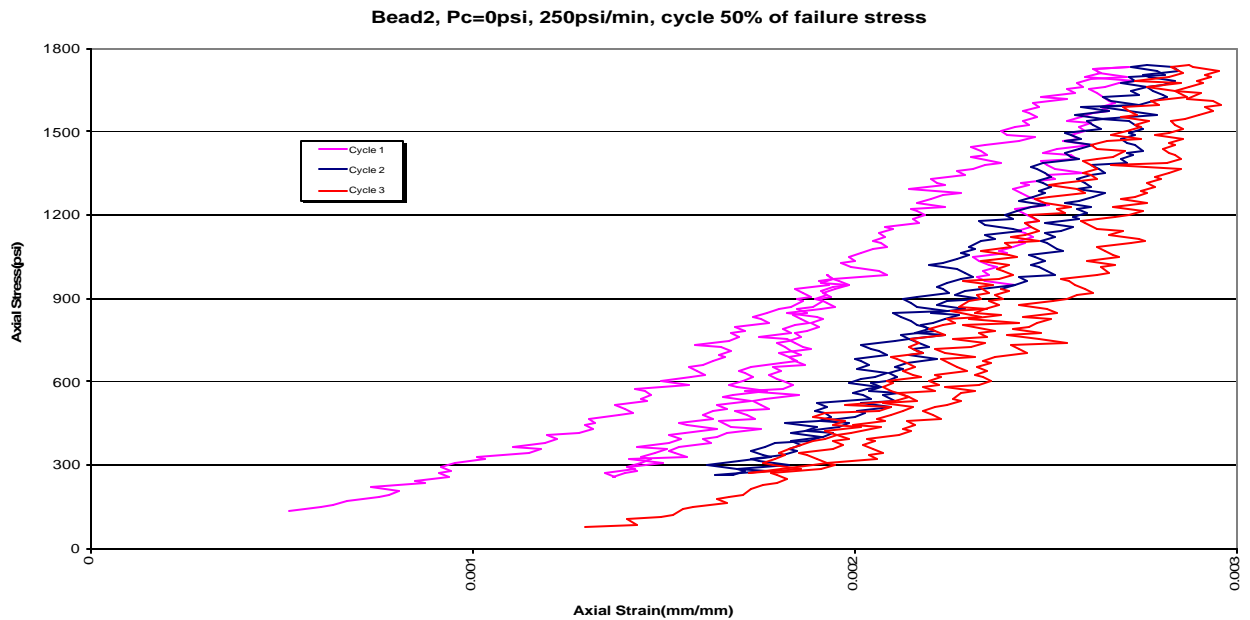


**Figure B33—Anelastic strain, cycled to 50% of failure load, for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**





**Figure B34—Anelastic strain, cycled to 50% of failure load, for bead slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**



**Figure B35—Anelastic strain, cycled to 50% of failure load, for foam slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

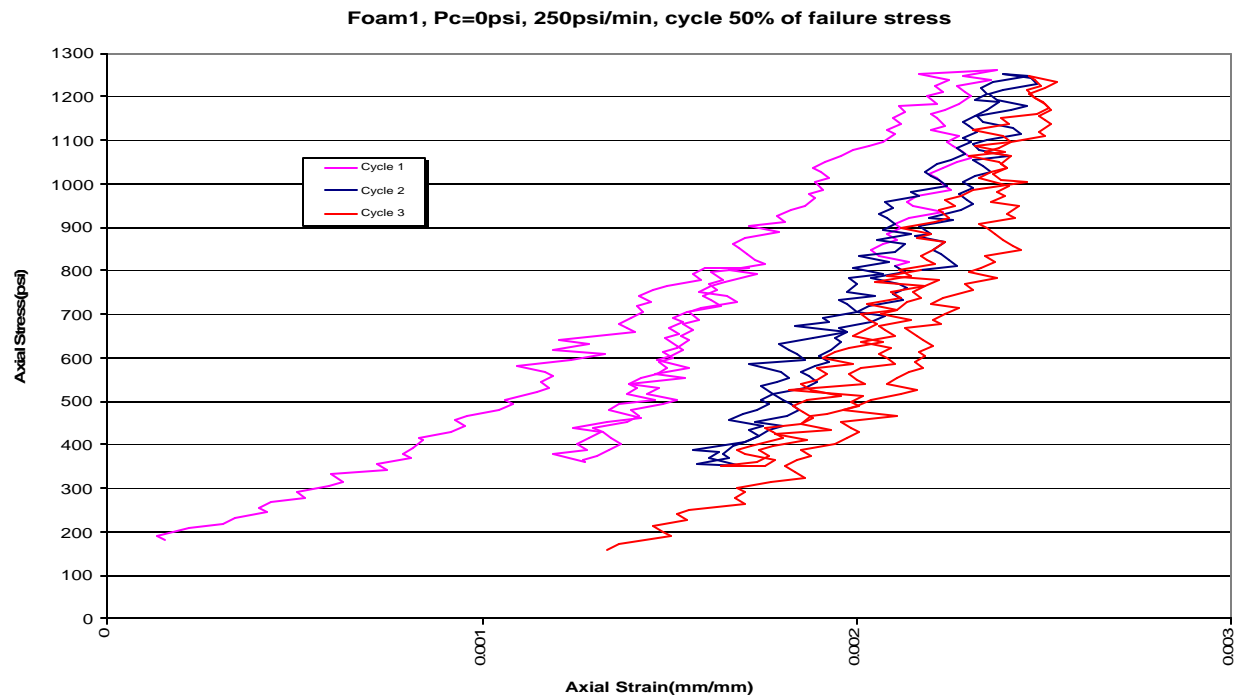




Table B1—Chronicle of 8ft Permeability Model Testing (mD)

Slurry #	Days Tested										
	1	7	14	23	37	44	51	60	63	65	66
1	0	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0.107	0.12	0.116	0.05	0.05
3	33	71	72	70	71	71	*	*	*	*	*
4	26	57	60	42	30	30	*	*	*	*	*
5	0	0	0	0	0	0	0	0	0	0	0
6	0	0	0	0	0	0	0	0	0	0	0
7	0	0	0	0	0	0	0	0	0	0	0
8	0	0	0	0	0	0	0	0	0	0	0

Slurry #	Days Tested										
	67	71	73	78	79	80	84	85	86	87	88
1	0	0	0	0	0	0	0	0	0	0	0
2	0.05	0	0.05	0.03	0.03	0.02	0	0	0	0	0
3	*	*	*	*	*	*	*	*	*	*	*
4	*	*	*	*	*	*	*	*	*	*	*
5	0	0	0	0.03	0.03	0.03	0.02	0.02	0.02	0.02	0.02
6	0	0	0	0	0	0	0.02	0.02	0.02	0.02	0.02
7	0	0	0	0	0	0	0.02	0.02	0.02	0.02	0.02
8	0	0	0	0	0	0	0	0	0	0	0

Slurry #	Days Tested							
	99	100	101	105	106	107	108	113
1	0	0	0	0	0	0.09	0.08	0.11
2	0	0	0	0.23	0.217	1.3	1.24	1.71
3	*	*	*	*	*	*	*	*
4	*	*	*	*	*	*	*	*
5	0	0	0	0	0	0	0	0
6	0.02	0.02	0.02	0	0	0.01	0	0
7	0.6	0.8	0.8	0.74	0.87	2.75	*	*
8	3.1	3.51	3.51	3.51	*	*	*	*

Day 1 Thru 44 - 100 PSI	Day 51 - 200 PSI	Day 60 Thru 73 - 300 PSI	Day 78 Thru 88 - 400 PSI	
Day 88 Thru 113 - 500 PSI				



Table B2—Chronicle of second set of 8ft Permeability Model Testing (mD)

Slurry #	Days Tested										
	1	2	3	4	7	8	9	10	11	14	15
1	2.41	3.05	3.81	4.7	5.08	5.59	5.59	5.71	5.71	5.71	5.84
2	0	0	1.23	1.23	1.23	1.15	1.22	1.21	1.22	1.22	1.27
3	0	0	0	0	0	0	0	0	0	0	0
4	0	0	2.29	1.4	1.4	1.52	1.4	1.4	1.4	1.4	1.27
5	0	0	0	0	0	0	0	0	0	0	0
6	6.73	4.82	8	8.63	9.65	9.52	9.52	9.65	9.65	8.89	9.01
7	0.89	0.76	1.78	2.03	2.41	2.29	2.67	2.67	2.67	2.67	2.67
8	0	0	0	0	0	0	0	0	0	0	0
9	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0

Slurry #	Days Tested										
	16	17	18	21	22	23	24	25	28	29	30
1	5.84	5.84	5.59	5.46	5.46	#	#	#	#	#	#
2	1.27	1.28	1.22	1.13	1.18	1.18	1.24	1.28	1.28	1.27	1.27
3	0	0	0	0	0	0	0	0	0	0	0
4	1.4	1.27	1.27	1.4	1.4	1.27	1.02	1.14	1.4	1.27	1.4
5	0	0	0	0	0	0	0	0	0	0	0
6	8.89	8.89	8.89	8.89	8.89	9.01	10.16	10.16	9.9	9.52	9.65
7	2.67	2.54	2.54	2.54	2.54	2.54	2.92	2.92	2.92	2.92	2.79
8	0	0	0	0	0	0	0	0.38	0.38	0.38	0.38
9	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0

Slurry #	Days Tested										
	31	32	37	39	43	45	50	56	60	66	71
1	#	#	#	#	#	#	#	#	#	#	#
2	1.26	1.11	1.29	1.27	1.24	1.26	1.27	1.26	1.27	1.27	1.27
3	0	0	0	0	0	0	0	0	0	0	0
4	1.4	1.4	1.4	1.27	1.27	1.27	1.27	1.4	1.4	1.4	1.4
5	0	0	0	0	0	0	0	0	0	0	0
6	9.9	9.9	9.9	10.79	12.44	13.97	15.62	17.52	18.28	25.77	27.04
7	2.92	2.92	2.79	3.05	3.05	2.29	2.92	2.92	2.92	3.17	3.17
8	0.63	0.63	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51
9	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0

All tested at 100psi

# denotes no longer testing





### Compositions for Table B2

Slurry # 1:	Type 1 + 20% Gel @ 12 ppg
Slurry # 2:	Type 1 + 18% Gel @ 12.5 ppg
Slurry # 3:	Type 1 + 16% Gel @ 13 ppg
Slurry # 4:	Type 1 + 3% SMS @ 12.5 ppg
Slurry # 5:	Type 1 + 2.5% SMS @13 ppg
Slurry # 6:	65:35 Type1:Poz + 16% Gel@12ppg
Slurry # 7:	65:35 Type1:Poz+ 12% Gel@12.5ppg
Slurry # 8:	65:35 Type1:Poz + 10% Gel@13ppg
Slurry # 9:	TXI LW + 2% SMS @ 12 ppg
Slurry#10:	TXI LW neat @ 13 ppg

Table B3—Chronicle of third set of 8ft Permeability Model Testing (mD)

Slurry #	Days Tested										
	1	2	3	5	8	10	12	15	18	20	23
1	7.36	6.86	7.11	6.86	6.73	6.73	3.55	5.71	7.11	6.6	4.57
2	8.63	6.35	10	5.84	6.09	7.49	3.94	5.71	7.24	6.6	6.09
3	2.29	3.05	3.17	3.17	3.3	3.3	0.89	2.92	3.55	3.81	3.43
4	0	0	0	0	0	0	0	0	0	1.27	1.27
5	0	0	0	0	0	0	0	0	0	0	0
6	0	0	0	0	0	0	0	0	0	0.38	0.38
7	5.97	5.97	6.86	7.24	6.73	6.98	4.7	5.84	7.11	6.98	6.22
8	32.1	34.2	36.2	35	35.6	35.8	31.2	x	x	x	x
9	50.5	53.3	57.4	56.8	56.8	57	50.3	x	x	x	x
10	35.9	36.2	37.8	37.5	38.1	38.1	35	x	x	x	x

Slurry #	Days Tested										
	26	28	30	33	39	44	54	60	69	73	82
1	6.73	5.71	6.60	6.60	6.86	6.86	6.6	3.68	3.55	3.3	2.41
2	6.09	6.35	6.09	6.60	6.86	6.6	6.86	6.98	7.62	7.11	7.62
3	6.86	5.33	6.35	8.63	12.70	8.00	9.27	15.87	18.79	19.81	x
4	1.27	1.52	2.03	4.57	16.00	13.94	14.47	4.57	6.6	8.76	21.58
5	0	0	0	0	0	0	0	0	0	0	0
6	0.63	0.51	0.51	0.51	0.51	0.51	0.51	1.02	0.63	0.63	0.51
7	6.47	6.73	6.60	7.11	7.36	6.22	4.95	7.74	8.38	8.63	10.28
8	x	x	x	x	x	x	x	x	x	x	x
9	x	x	x	x	x	x	x	x	x	x	x
10	x	x	x	x	x	x	x	x	x	x	x



Slurry #	Days Tested										
	92	113									
1	3.3	3.3									
2	9.14	12.32									
3	X	X									
4	X	X									
5	0	0									
6	0.63	0.51									
7	19.14	X									
8	X	X									
9	X	X									
10	X	X									

### Compositions for Table B3

Slurry # 1:	Type 1 + 2% SMS @ 13.4 ppg
Slurry # 2:	Type 1 + 2% SMS @ 13 ppg
Slurry # 3:	TXI LW + 3% SMS @ 11 ppg
Slurry # 4:	TXI LW + 3% SMS @ 11.5 ppg
Slurry # 5:	65:35 Type1:Poz + 6% Gel @13.5 ppg
Slurry # 6:	50:50 Type1:Poz + 6% Gel @ 13.4 ppg
Slurry # 7:	50:50 Type1:Poz + 8% Gel @ 12.8 ppg
Slurry # 8:	50:50 Type1:Poz + 10% Gel @ 12.4 ppg
Slurry # 9:	TXI "H" + 12% Gel @ 12 ppg
Slurry #10:	TXI "H" + 8% Gel @ 12.5 ppg

<sup>1</sup> API Recommended Practice 10B: "Recommended Practice for Testing Well Cements," 22nd Edition, American Petroleum Institute, Washington, D.C., December 1997.

<sup>2</sup> ASTM C469, Standard Test Method for Static Modulus of Elasticity (Young's Modulus) and Poisson's Ratio of Concrete in Compression.

<sup>3</sup> "Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens," ASTM C496-96, West Conshohocken, PA, 1996.